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RCRA FACILITY INVESTIGATION QUALITY ASSURANCE PROJECT PLAN PHASE III SOILD SOLID WASTE MANAGEMENT UNIT 7 (SWMU7) VOLUME II APPENDICES WITH TRANSMITTAL NSA CRANE IN

10/1/2000

TETRA TECH

SWMU 7
Phase III Soils RFI
QAPP

Naval Surface Warfare Center
Crane Division
Crane, Indiana

Volume II - Appendices

Southern Division
Naval Facilities Engineering Command
Contract Number N62467-94-D-0888
Contract Task Order 0056

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APPENDIX A

ORR and DEMO SOILS RFI CHRONOLOGY

DETAILED DISCUSSION OF CURRENT ORR & DR STATUS

The current status with regard to completing the Soils Resource Conservation and Recovery Act (RCRA) (RFI) requirement is the result of a long chain of events (described below) leading to a focused list of target analytes in soils at the ORR and the deferral of actions at the DR Navy. To understand how the list of parameters for soils was developed and subsequently narrowed down and why action at the DR Navy was deferred, it is necessary to understand what steps have been completed and the order in which they were implemented. Figure A-1 depicts these major events in chronological order. Some of these events relate to ground water investigations, which were useful for the interpretation of soils contamination.

Subsections labeled "ID1 through ID16" (ID = Task Identification) refer to the Task Name shown in Figure A-1.

ID1 SOIL SAMPLING FOR PHASE II RFI - ORR (USACEWES)

The United States Army Corps of Engineers Waterways Experiment Station (USACEWES) conducted an RFI soil investigation at the ORR. Soil samples were obtained from thirteen (13) test borings in August of 1990 for physical characterization and chemical pollutant identification. Soil samples were taken from specific layers within the borings; 0.2 –0.5 feet bgs, 3-6 feet bgs, 12-18 feet bgs, 18-24 feet bgs, and within 6 feet of the ground water table. These samples were analyzed by the USACE laboratory for explosives, inorganics, volatile and semivolatile organics. Soil contamination was found and reported.

ID2 ORR SOILS PHASE II RFI DRAFT REPORT (USACEWES)

The Draft RFI Phase II Soils Report for the ORR was issued in April 1991(this report did not include any part of SWMU 6 – DR). The concluding two sentences of the Executive Summary state "No RFI Phase III Soils study is needed at the Old Rifle Range. The remediation of the soils in the burn pits should be included as part of the closure plan for the Old Rifle Range burning unit." (UASCEWES, 1991). Based on review of available information, this Draft RFI Phase II Soils Report for the ORR appears not to have been finalized.

ID3 DRAFT CCCRA WORK PLAN FOR ORR, DR & ABG - JUNE 1993

Rust Environment & Infrastructure (RE&I) as a subcontractor to Brown & Root Environmental (B&RE is the predecessor of TtNUS) prepared a Draft Current Contamination Conditions Risk Assessment (CCCRA) Work Plan in June of 1993. This Work Plan addressed the sampling and risk assessment needs for the Ammunition Burning Grounds (ABG), ORR, and DEMO. Prior to this, although the ABG and ORR had been investigated, soil sampling and analyses had not been conducted at the DEMO.

ID4 FINAL CCCRA WORK PLAN JULY 1995 (RE&I)

RE&I, as a subcontractor to B&RE, prepared the Final CCCRA Work Plan in July of 1995. This Work Plan addressed comments received from the U.S. EPA on the draft CCCRA. No additional soils sampling had been conducted at the ORR or DEMO since the draft version of the Work Plan was prepared (described in Section ID3). More data was required to complete the Final CCCRA.

ID5 ORR AND DR SAMPLING - AUGUST 1995 (RE&I)

RE&I implemented the provisions of the 1995 CCCRA Work Plan and collected the data required for the CCCRA. Six surface soil samples (0-2' bgs) were collected and analyzed for inorganics, semivolatile organics, and explosives.

At the time the CCCRA Work Plan was prepared, there had been no soil investigation conducted at the DEMO. RE&I recommended that eight (8) surface soil composite samples (0-2 feet bgs) be taken from the Army Demolition Area (DR Army) and three surface soil composite samples be taken from the Navy Demolition Area (DR Navy). In addition, three background surface soil samples were recommended. All 14 soil samples were taken in 1995 and analyzed by Southwest Laboratories for explosives, inorganics and semivolatile organics.

ID6 DRAFT RFI FOR GROUND WATER SWMU 6&7 NOVEMBER 1995 (USACEWES)

This document addresses ground water investigations at SWMUs 6&7 from November 1989 to December 1992. These studies become relevant to the DR Navy soil investigation outlined in this QAPP with regard to the manganese hot spot. The purpose was to determine the presence or absence, the nature, the rate and extent of migration, and the concentrations of hazardous constituents that may have been released into ground water from activities conducted at the DEMO and the ORR.

Conclusions presented in the draft report were that metals, cyanides, sulfides, and nitrates were detected in significant and verifiable quantities in monitoring wells at the DEMO. A localized area at the DR Navy exhibited significantly elevated concentrations of some metals and was referred to as a ground water hot spot. Organics other than explosives were not present in significant or verifiable quantities in three rounds of sampling and analysis of ground water from monitoring wells at the DR and ORR.

ID7 EPA DATA VALIDATION MEMO FOR ORR, DR & ABG - 1997

Following preparation of the CCCRA Work Plan, one of the first tasks conducted by RE&I was a trend analysis of the data collected to that date. As part of this effort it was discovered that very little of the analytical data had undergone a data validation effort of sufficient rigor to produce results that meet the minimum data usability standards for risk assessment purposes. This issue was brought to the attention of U.S. EPA Region 5, whereupon a relatively detailed review of select datasets from each laboratory was performed by U.S. EPA Region 5 and their contractor (A.T. Kearney) (U.S. EPA, 1997).

Based on the data validation memorandum issued by U.S. EPA Region 5 (U.S. EPA, 1997), all of the non-explosive soils data at the ORR from the Corps of Engineers laboratory were rejected because of incomplete QC documentation. With respect to this environmental medium, the remaining database for soil at the ORR consisted of the 1990 explosives data by the Corps of Engineers laboratory (43 surface and subsurface soil samples) and the six surface soil samples collected by RE&I in 1995. The Corps of Engineers did not collect any soil samples at the DR Navy.

Based on the data validation memorandum issued by U.S. EPA Region 5 (U.S. EPA, 1997), all of the analytical data for soils at the DEMO were valid and therefore acceptable for use in the CCCRA.

ID8 ORR FOLLOWUP SAMPLING 1997 (RE&I)

Based on RE&I's assessment of the valid data remaining after the U.S. EPA Region 5's review, five additional samples were proposed for collection at the ORR to complete the database for risk assessment. These samples were collected and made part of the complete analytical database for the Risk Assessment.

ID9 DRAFT CURRENT CONTAMINATION CONDITIONS RISK ASSESSMENT REPORT NOVEMBER 1997

Based on all of the data collected as previously described (less the rejected data), RE&I prepared the draft Current Contamination Conditions Risk Assessment Report (B&RE, 1997). The chemicals of concern (COCs) and footnotes indicating the critical pathway by SWMU were as follows:

Chemical of Concern	ORR,	DR Navy,	DR Army,

	SWMU 7	SWMU 6	SWMU 6
Metals			
Aluminum		X(1)	
Arsenic	X(1)	X(1)	X(1)
Beryllium	X(3)	X(1&2)	
Manganese	X(1)	X(1)	X(1)
Nickel		X(1)	
Energetics			
2,4,6-Trinitrotoluene	X(1)		
2,6-Dinitrotoulene	X(1)		
RDX	X(1)	X(1)	
Polycyclic Aromatic Hydrocarbons			
Benzo(a)anthracene	X(1)		
Benzo(a)pyrene	X(2)		
Benzo(b)fluoranthene	X(2)		
Dibenzo(a,h)anthracene	X(2)		
Indeno(1,2,3-cd)pyrene	X(2)		
Organochlorine Pesticides			
Heptachlor Epoxide	X(1)		

1 Based on ingestion of ground water

2 Based on ingestion of beef and milk

3 Based on dermal contact with ground water

ID10 SITE VISIT FOR PHASE III SOILS RFI - MARCH 1998 (TtNUS)

A site visit was conducted on March 4, 1998 for the purpose of identifying information needed to complete the RFI Work Plan for soils at SWMU 6 (DR Navy) and SWMU 7 (ORR). At the time of this visit, both the Ground Water RFI and CCCRA were in draft form, identifying areas of ground water contamination, critical pathways, receptors, and chemicals of concern.

Highlights of the visit by SWMU are described in the following paragraphs.

SWMU 6 - Demolition Range

Information Review and Site Reconnaissance

Prior to conducting the site reconnaissance, and as part of work plan preparation (see ID13), aerial photographs of the DEMO dated 8-1-48 were reviewed. Other than the chemical data collected by RE&I

in 1995, no historical chemical (analytical) data could be found. The pre-1995 data for SWMU 6 was primarily geotechnical data (Dunbar, 1982), which consisted of soil classifications, grain-size distribution, etc.

The team conducted a walk-over at the northern end of the DR Army (south ridge) site. The general locations of the background soil samples collected by RE&I were noted. The team then walked to the DR Navy (east ridge) site. This area is not used at the same frequency as the south ridge, however it is still active. The issue associated with this site is that there is an apparent contamination hot spot based on previous analysis of ground water data (manganese). Data needs are presented in the following paragraphs.

Soil Data Needs

It was confirmed by the Environmental Site Manager (ESM) that the primary focus of soil investigation at SWMU 6 is to better define the manganese hot spot at the DR Navy. A secondary focus was to evaluate the need for additional background data. However, the Base-Wide Background Soil Investigation currently underway should generate data sufficient for comparative purposes. It was acknowledged that additional data needs may also arise after a more detailed review of the aerial photos was completed.

SWMU 7 - Old Rifle Range

Information Review and Site Reconnaissance

Prior to conducting the site reconnaissance, aerial photographs dated 8-1-48 were reviewed to examine past rifle (and pistol) range operations and the potential for range activities just north of the current ORR. During the initial project teleconference, it was agreed that there may have been range activities conducted just north (and across the stream of the "maintained" area) of the current ORR. This recently identified area is commonly referred to as the Old Pistol Range (OPR). To date, no environmental samples have been collected for laboratory analyses from the OPR.

During the site reconnaissance, the team parked at the north end of the ORR and walked down into the hollow at the entrance to the OPR. This area had the remnants of a small wooden structure (possibly a shooting enclosure), approximately three 55-gallon drums (probably used for spent cartridges or other refuse) and DANGER signs delineating the presence of a pistol range. A backstop consisting of mounded earth and metal target holders used to accumulate fired projectiles had been constructed roughly 100 yards upstream of the apparent location of the firing line. Additionally, it appeared that firing took place into the side of the hill to the west of the firing line. Data needs are presented in the following paragraphs.

Soil Data Needs

It was confirmed by the ESM that soil data are needed to characterize potential contamination in the soils of the OPR backstop, the hillside to the west of the pistol range firing line, soils close to the 55-gallon drums, and the upper reaches of the hillside behind the final concrete-reinforced backstop/target mechanism located on the south end of the rifle range. Also, additional soil samples may be needed on, and at the base of the berms in the main area of the range. Additional background samples also need to be obtained for the ORR and upgradient from the OPR for comparison with site chemical concentrations to evaluate exceedances of background concentrations. The Base-Wide Background Soil Investigation currently underway should generate data sufficient for comparative purposes.

ID11 FINAL RFI FOR GROUND WATER SWMU 6&7 JUNE 1998 (USACEWES)

This was the final version of the document prepared to address ground water investigations at SWMUs 6&7 from November 1989 to December 1992.

Conclusions presented in the final version with regard to the DR were identical to those presented in the draft report. Metals, cyanides, sulfides, and nitrates were detected in significant and verifiable quantities in monitoring wells at the DEMO. Organics other than explosives were not present in significant or verifiable quantities in three rounds of sampling and analysis of ground water from monitoring wells at the DR and ORR.

ID12 FINAL CURRENT CONTAMINATION CONDITIONS RISK ASSESSMENT REPORT FEBRUARY 1999

Following incorporation of U.S. EPA Region 5 comments, RE&I prepared the final Current Contamination Conditions Risk Assessment Report (TtNUS, 1999a). The COCs and critical pathways with regard to the two SWMUs in this Work Plan were identical to those identified in the draft CCCRA Report (see ID9).

ID13 DRAFT SECTION 1 FOR SOILS RFI QAPP SUBMITTED TO U.S. EPA APRIL 23, 1999 (TtNUS)

Following the issuance of the final CCCRA and the confirmation that COCs for these SWMUs were identified, the pre-QAPP process with U.S. EPA Region 5 for the soils RFI was initiated by phone.

TtNUS prepared the draft Section 1 of the Soil RFI QAPP, which included a review of what had been conducted to date on the project, the regulatory scenario, historical data issues, the CCCRA, and other facts pertinent to data quality objective (DQO) development. The draft Section 1 was submitted on April 23, 1999 to the U.S. EPA Region 5 and Navy representatives, and was used as a point of discussion during the pre-QAPP teleconference.

ID14 PRE-QAPP TELECONFERENCE WITH U.S. EPA TO DISCUSS DRAFT SECTION 1 MAY 4, 1999

Prior to constructing the QAPP, U.S. EPA Region 5 requires that Section 1 of this document be prepared and reviewed by the agency. This allows an opportunity for the agency to refine and agree to the DQOs prior to the generation of the draft document.

Section 1 of this QAPP was prepared and submitted to the U.S. EPA Region 5. A pre-QAPP teleconference was held on May 4, 1999 to discuss the draft Section 1 of the RFI QAPP (U.S. EPA, 1999).

The primary outcome of the teleconference was a consensus that the 14 COCs as identified in the CCCRA would be the only chemical parameters evaluated as part of this RFI. U.S. EPA indicated that all concerns regarding other contaminants had been addressed by virtue of the completed CCCRA conducted by RE&I. Another significant outcome was consensus that the Navy would be responsible for deciding whether interim remedial measures would be warranted at the DR Navy based on the results of the Phase III RFI. A final outcome was agreement that no additional data collection is necessary at the DR Army.

Specific conclusions for each site were as follows:

The COCs reported in the CCCRA (TtNUS, 1999a) for SWMUs 6 and 7 were narrowed down per site as shown in the table that follows. The DR Army area is not included because U.S. EPA indicated they were satisfied that they had all of the soils results needed at this time. The primary COC at SWMU 6 is manganese because it is associated with the hot spot issue.

Chemical of Concern	ORR, SWMU 7	DR Navy, SWMU 6
Metals		
Aluminum		X
Arsenic	X	X
Beryllium	X	X
Manganese	X	X[1]
Nickel		X
Energetics		
2,4,6-Trinitrotoluene	X	
2,6-Dinitrotoulene	X	
RDX	X	X
Polycyclic Aromatic Hydrocarbons		
Benzo(a)anthracene	X	
Benzo(a)pyrene	X	
Benzo(b)fluoranthene	X	
Dibenzo(a,h)anthracene	X	
Indeno(1,2,3-cd)pyrene	X	
Organochlorine Pesticides		
Heptachlor Epoxide	X	

1 Manganese is the primary COC with regard to the hot spot issue

SWMU 6 – Demolition Range

Extent of contamination at SWMU 6 is not an issue as it has been addressed satisfactorily by other activities, including previous sampling and the completed CCCRA. The only outstanding issue at SWMU 6 is further characterization of the manganese hot spot. TtNUS was assigned the responsibility of evaluating the ground water data for wells near the alleged hot spot and making recommendations for additional investigation. It was suggested that this should include developing the wells in question prior to identifying soil sample locations (if needed).

SWMU 7 – Old Rifle Range

Extent of contamination is a primary issue at the ORR and, more specifically, at the portion of the SWMU that has recently been identified as having pistol shooting operations (the OPR located to the north of the ORR proper). The QAPP will address this issue.

ID15 EPA APPROVAL OF USACEWES FINAL RFI FOR GROUND WATER SWMU 6&7 JUNE 8, 1999

U.S. EPA Region 5 approved the RFI for Ground Water at SWMU 6&7. U.S. EPA Region 5 restated (U.S. EPA, 1999) the DR Navy metals contamination conclusion in the approval letter and indicated that "the U.S. Navy recently proposed an investigation to define the accuracy of these findings, and to delineate the hot spot if confirmed at the Demolition Range." With regard to the ORR, the approval letter goes on to state, "They (the US Navy) are planning an RFI for soil at the backstop areas which were not previously investigated since they were considered 'inactive ranges'." This Phase III Soil RFI QAPP addresses the proposed soil investigations in both of these areas.

ID16 MANGANESE HOT SPOT ISSUE PAPER - AUGUST 11, 1999 (TtNUS)

As requested in the May 4, 1999 teleconference with EPA, TtNUS performed an evaluation of ground water analytical results at the DEMO (SWMU 6). Previous results indicated the presence of elevated concentrations of some metals in the Navy Explosive Ordnance Disposal (EOD) area (DR Navy). The TtNUS evaluation was performed to confirm the accuracy of these findings and to delineate this localized area of contamination (referred to as the manganese hot spot).

Ground water samples collected from wells screened within the Upper Pennsylvanian-age aquifer in the Navy EOD area of the DEMO showed elevated concentrations of various metals. Unusually high concentrations of vanadium, cadmium, beryllium, zinc, nickel, aluminum, arsenic, manganese, cobalt, iron, and magnesium occurred in three shallow wells monitoring the Pennsylvanian-age rocks. Three wells within this area, namely 06-06, 06-07, and 06-12, are within 300 feet of each other and exhibit consistently elevated concentrations of these metals. This indicates that the uppermost ground water in that isolated area is being contaminated by a local source. Low pH (high acidity) and high conductivity accompany the anomalously high levels of metals. Murphy and Wade (1998) with the USACEWES suggested that additional sampling of surface and shallow soils and continued monitoring of these wells was necessary to confirm contamination and to delineate its source.

Based on the TtNUS evaluation of the ground water hot spot issue and discussions with Base personnel, the USACEWES, representatives of the U.S. EPA, and Southdiv representatives for NSWC Crane, numerous activities were proposed as part of the Hot Spot Letter Report (TtNUS, 1999b). The following is a summary of the stated investigative needs at that time with regard to the hot spot issue.

- Wells 06-06, 06-07, 06-12, 06C01P3 and 06-01A should be redeveloped and resampled for the five DR Navy COC metals (manganese, aluminum, arsenic, nickel, and beryllium).
- Standard water quality parameters should be collected including temperature, pH, specific conductance, and turbidity.
- Ground water samples should be collected using low-flow sampling techniques.
- Samples should be collected for unfiltered laboratory analyses.
- Results should be statistically analyzed and compared to appropriate screening levels.
- If elevated concentrations of metals are detected in these samples, a geophysical survey should be considered in the immediate area of the elevated metals to determine if a local source of contamination is indicated in the shallow subsurface. Pending the results of this survey, additional delineation should be considered and soil borings may be installed to confirm the source area.

ID17 DRAFT SWMU 6 & 7 PHASE III SOILS RFI QAPP SUBMITTED - FEBRUARY 23, 2000 (TtNUS)

Following preparation and review by the Navy, TtNUS finalized and submitted the Draft SWMU 6 & 7 Phase III Soils RFI QAPP for review by US EPA Region 5.

ID18 EPA COMMENTS ON THE DRAFT SWMU 6 & 7 PHASE III SOILS RFI QAPP - APRIL 20, 2000

EPA reviewed and provided comments on the Draft SWMU 6 & 7 Phase III Soils RFI QAPP. Many of the comments related to the statistical basis for determining the potential manganese hot spot at SWMU 6. A total of 21 comments were prepared by EPA requesting clarifications of certain issues and other editorial comments.

ID19 RESPONSE TO COMMENTS ON THE DRAFT SWMU 6 & 7 PHASE III SOILS RFI QAPP - JUNE 6, 2000

TtNUS prepared responses for each comment, which were reviewed by the Navy and then forwarded to EPA.

ID20 RESPONSE TO COMMENTS TELECONFERENCE WITH EPA ON THE DRAFT SWMU 6 & 7 PHASE III SOILS RFI QAPP - JUNE 27, 2000

Following EPA's review of the responses to comments, EPA along with IDEM requested that a teleconference be held on June 27,2000 to discuss certain issues. Because this teleconference was critical to the EPA's final decision regarding required actions at SWMU 6, a detailed summary is provided below.

The following organizations and representatives were in attendance:

NSWC Crane - Mr. Tom Brent
State of Indiana (IDEM) - Mr. Doug Griffin
EPA Region 5 - Mr. Peter Ramanauskas and Dr. Arthur Lubin
Tetra Tech NUS (TtNUS) - Dr. Roger Clark; Mr. Mark Francis; Dr. Tom Johnston; and Mr. Brian Lewis

- IDEM questioned if kriging was being done in support of risk assessment. TtNUS affirmed that the risk assessment on the DRNavy had already been performed so this investigation did not include a risk assessment.

- IDEM asked why the nature and extent of manganese contamination hadn't been determined during risk assessment. TtNUS explained that manganese was identified as a COC in the risk assessment. Carol Witt-Smith (previous EPA regulator) had suggested a possible manganese 'hot spot' based on high groundwater concentrations at the site.

- TtNUS stated within the Draft Work Plan that they planned on resampling the groundwater wells to determine whether evidence for a hot spot exists now, then placing a grid with no more than 25 soil sampling locations near the groundwater wells that are statistically higher than upgradient. Kriging of soil data would then be used to determine the 'hot spot' boundary. IDEM asserted that it must first be determined how big of a 'hot spot' was being looked for and what would be done if one was found - before deciding on the sampling strategy.

- The basis for determining the number of samples required to detect a hot spot in DRNavy soils was discussed. The group agreed that no one knew the size of the hot spot or whether the hot spot has a diffuse boundary. NSWC indicated that the "hot spot" could be a localized region of elevated background or it could be a bomb casing of some sort. IDEM explained the difficulty in trying to estimate the number of soil samples required for locating a hot spot in an area of unknown size when the ground water data are not yet available from the DRNavy wells. EPA acknowledged that the number of samples required to find a hot spot with a well-defined boundary could approach 500,000, which was an unrealistic number of samples. Ultimately, TtNUS' proposition to collect samples at up to 25 locations was not disputed.

- TtNUS consulted the risk assessment report and determined that the pertinent risk receptors for the DRNavy were through groundwater consumption and that hazard indices (HI) values were 5 to 16. IDEM maintained that because closure wouldn't occur for ~ 50 years and that ongoing operations at the facility may cause additional contamination, institutional controls to restrict access to the groundwater should be implemented on the subpart X permit until the closure plan takes place. It was suggested that groundwater samples would be taken to determine if manganese is present above upgradient levels. IDEM agreed that this would probably be a good strategy to follow. If so, institutional controls would be implemented and action would be deferred until closure.

- IDEM asserted that the HI values ranging from 5 to 16 were significant. However TtNUS also advised IDEM that operations were not scheduled to cease for at least another 50 years. IDEM felt that quantifying the groundwater manganese concentrations was warranted, but that actions to be taken might be tempered by the realism of whether groundwater would actually be consumed at this location.

- EPA agreed to look at the permit to determine whether the DRNavy "remediation" (if required) could be handled as part of the closure plan for the Subpart X permit for the DR (which includes the DRArmy and DRNavy). It was suggested that TtNUS would collect the recommended groundwater samples to determine whether there appeared to be a hot spot in groundwater. If EPA's determination on the permit indicated that the DRNavy ***can*** be covered under the closure plan, any actions with regard to manganese hot spot remediation would probably be addressed when the facility closes. If EPA's determination on the permit indicated that the DRNavy ***cannot*** be covered under the closure plan, any actions with regard to manganese hot spot remediation would have to be controlled by EPA and IDEM at that time.

- For the DRNavy area only, TtNUS asked if the soil sampling portions of the Work Plan would need to be removed and IDEM indicated that EPA might be able to “conditionally approve” the document excluding all soil sampling sections. Final disposition regarding this matter would be discussed following the determination by EPA regarding closure applicability for the DRNavy.

- The State questioned the use of the "20x" rule when comparing total analysis data to TCLP limits. TtNUS explained the use and volunteered to include more explanation in the text of the QAPP.

- Action items for the teleconference included: EPA - review Crane permit to determine whether DRNavy is covered by the subpart X permit; TtNUS - add justification for using the "20x" rule on TCLP limit comparisons for determining nature of IDW.

ID21 EPA DECISION TO REMOVE SWMU 6 FROM THE PHASE III SOILS RFI QAPP - SEPTEMBER 20, 2000

NSWC Crane received a letter from EPA with a decision regarding SWMU 6. The following paraphrases the critical elements of EPA’s decision:

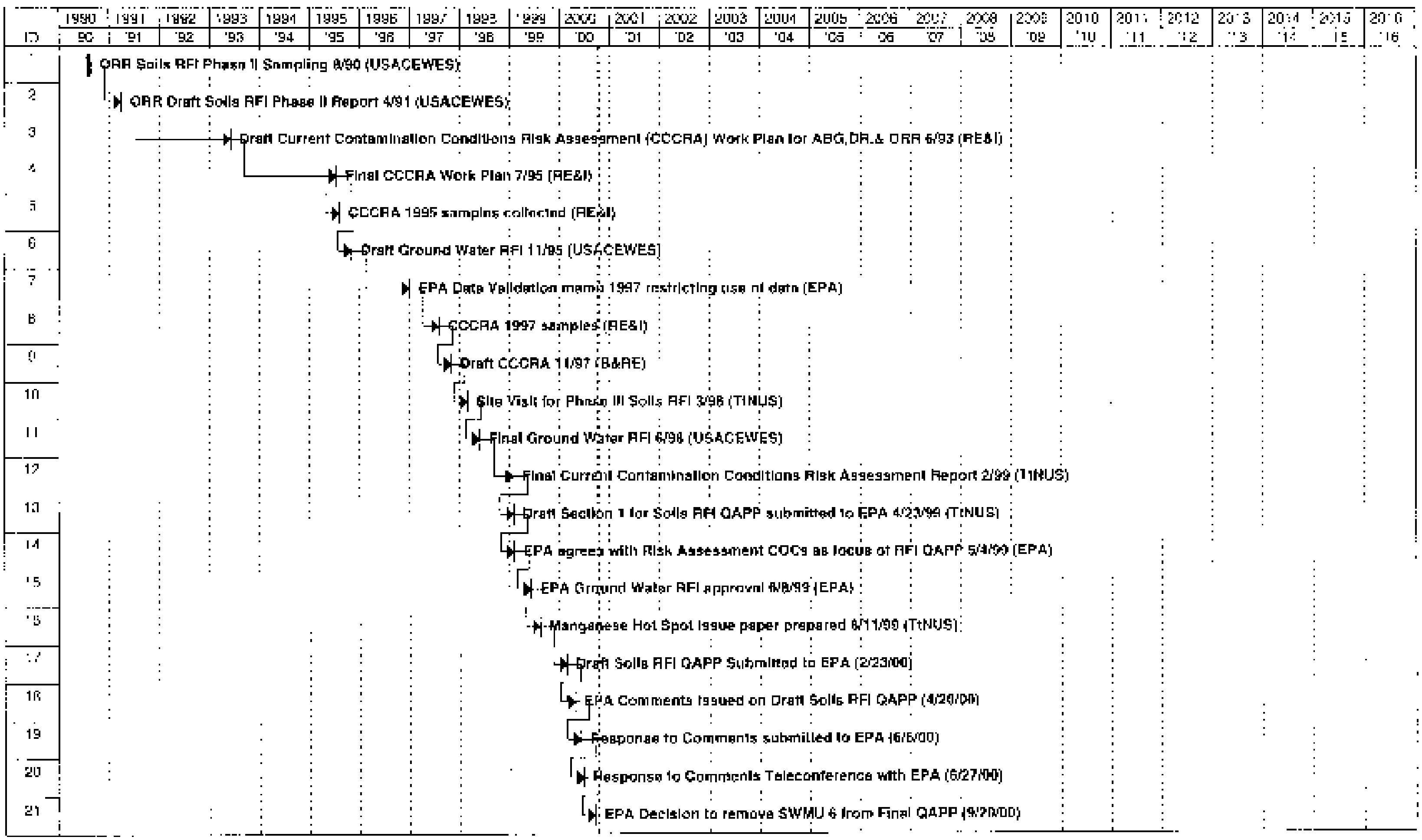
> The Navy may disregard all sections in the Draft Solid Waste Management Unit (SWMU) 6 & 7 Phase III Soils RCRA Facility Investigation (RFI) Quality Assurance Project Plan (QAPP) dated February 2000 that relate to the ground water sampling at SWMU #6. The purpose of its inclusion in the RFI was to investigate the presence of a potential manganese “hot spot” in the soils of the Demolition Range (DR) Navy.
>
> The Demolition Range (SWMU #6) is a regulated open detonation unit under the RCRA Subpart X permit issued in November 1999. This unit has been subdivided into the DR Army and the DR Navy. It has also been designated as SWMU #6 and has been undergoing corrective action investigations.
>
> As part of the work involved in permitting the unit, the DR underwent a risk assessment along with the other open burning/open detonation units at NSWC (i.e., Ammunition Burning Grounds and Old Rifle Range). The risk assessment identified risks from two aquifers at the DR. The constituents of concern were identified as: Aluminum, Arsenic, Beryllium, Manganese, Nickel, and RDX. No risks were identified in the DR soils.
>
> A conference call was held on June 27, 2000 between the Navy, Tetra Tech NUS, Inc., the Indiana Department of Environmental Management, and the USEPA to discuss the draft RFI QAPP developed for SWMUs 6 & 7. A question arose as to the practical benefits of the manganese “hot spot” study at the DR Navy at this time because soils were not identified as a risk driver in the risk assessment. Soils

contamination at the DR Navy can be addressed via the RCRA Subpart X permit at unit closure. The DR unit is a permitted open detonation unit with an estimated closure date of 2015.

Because the risk assessment identified groundwater as the sole risk pathway at the DR, groundwater is being monitored under the Subpart X permit and groundwater use has been restricted. The groundwater use restriction eliminates the risk pathway of groundwater ingestion. The Navy is required to perform semi-annual RCRA Subpart F groundwater detection monitoring at the DR under the permit. The groundwater wells used in the risk assessment study done at the DR are being monitored under the Subpart X permit. Although wells having elevated manganese levels specific to the DR Navy that were identified through previous RFI work are not part of the DR point-of-compliance well network, the DR Navy is located within the point-of-compliance. Previous RFI investigations indicate that groundwater seeping from the DR Navy would be collected by the NPDES permitted sedimentation ponds around the perimeter of the DR or, because the aquifer underlying the DR Navy is discontinuous, migrate downward through fractures in the bedrock to be intercepted by the point-of-compliance wells.

It is the recommendation of this office that the proposed manganese “hot spot” study may be deferred until closure of the DR, if required at that time. The Navy should remove all reference to the groundwater sampling at SWMU #6 and may proceed with revision of the RFI QAPP to address the Old Rifle Range (SWMU #7). The Navy will need to add Aluminum to the groundwater monitoring program currently in place at the DR. By way of this letter, it is understood that all sections of the RFI QAPP pertaining to the DR will be disregarded and remediation at the DR will be addressed under unit closure or if groundwater monitoring detects contaminant migration requiring corrective action under the Subpart X permit.

Preparation of this QAPP (final version) concludes the chronology as of the date of this writing.



DRAWN BY: L. LANEY DATE:	Tetra Tech NUS, Inc.	CONTRACT NUMBER 7829	OWNER NUMBER 0056
CHECKED BY: M. FRANCIS DATE:	SEQUENCE OF EVENTS FOR DEMOLITION RANGE AND OLD RIFLE RANGE NSWC CRANE, CRANE, INDIANA	APPROVED BY MRF DATE 2/24/00	
COST/SCHEDULE AREA		APPROVED BY RAC DATE 2/24/00	
SCALE AS NOTED		DRAWING NO. FIGURE A-1	REV 1

P:\GIS\NSWC CRANE\7829.APR FIGURE 8 TEMPLATE

APPENDIX B

FIELD STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE

NUMBER CTO 56-1

BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A HAND AUGER OR GEOPROBE®

STANDARD OPERATING PROCEDURE
NUMBER CTO 56-1

BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A HAND AUGER OR GEOPROBE®

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for advancement and soil sampling using a hand auger or Geoprobe® for the Phase 3 Soil RFI for SWMU 7, NSWC Crane.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Non-latex Gloves
Cotton Gloves
Writing Utensil
Boring Log Sheets: A copy of this form is included in Appendix B, SOP CTO 56-4.
Soil Sample Log Sheets: A copy of this form is included in Appendix B, SOP CTO 56-4.
Complete Hand Auger Assembly (stainless steel bucket bits, a series of extension rods (available in 2', 3', 4' and 5' lengths, and a "T" cross handle).
Geoprobe® or Equivalent DPT Equipment
Geoprobe® Sampling Kit
Stainless-steel Mixing Bowls
Disposable Plastic Trowel or Stainless-steel Trowel
Required Sample Containers with Labels, Tags and Appropriate Preservative: A copy of sample sample labels and tags are included in Appendix B, SOP CTO 56-4. All sample containers for analysis by fixed-base laboratories will be supplied and deemed certified clean by the laboratory.
Required Decontamination Materials

3.0 PROCEDURES FOR ADVANCEMENT OF BOREHOLE AND SOIL SAMPLING USING A HAND-HELD BUCKET AUGER

3.1 Clear the area to be sampled of any surface debris (herbaceous vegetation, twigs, rocks, litter, etc.).

3.2 Don non-latex and/or cotton gloves and attach a properly decontaminated bucket bit to a clean extension rod and further attach the cross handle to the extension rod.

3.3 Begin augering by turning the “T” handle in a clock-wise fashion, thus turning the auger bit until the bucket bit is advanced approximately 6 inches into the soil. Add additional rod extensions as necessary to reach the desired depth.

3.4 After reaching the desired depth, slowly and carefully withdraw the bucket from the borehole.

3.5 Discard the top of core (approximately 1"), which represents any loose material collected by the bucket bit before penetrating the desired sample depth.

3.6 Utilizing the hand trowel remove the sample material from bucket bit into a properly decontaminated stainless-steel mixing bowl.

3.7 Log the recovered sample on the Boring Log Sheet (provided in Appendix B, SOP CT0 56-4).

3.8 Return the same bucket auger into the borehole and turn the auger as stated in step 3.3, advancing the auger bit an additional 6 inches into the soil (totaling 1 foot).

3.9 After reaching the desired depth, slowly and carefully withdraw the bucket from the borehole.

3.10 Discard the top of core (approximately 1"), which represents any loose material collected by the bucket bit before penetrating the desired sample depth.

3.11 Utilizing the hand trowel remove sample material from bucket bit into the same stainless-steel mixing bowl mentioned in step 3.6.

3.12 Log the recovered sample on the Boring Log Sheet (provided in Appendix B, SOP CT0 56-4).

3.13 Carefully remove gravel, vegetation, roots, twigs, litter, etc. from the sample.

3.14 Composite sample, if required. After completing steps 3.1 through 3.13 above, move to the next borehole location that will make-up the composite sample and repeat steps 3.1 through 3.13. Note: a composite sample exists as a combination of more than one sample at various locations and/or depths and times, and are not to be collected for volatile organic analysis. After all samples to be composited have been collected, mix equal volumes of soil from each of the aliquots and continue with step 3.15.

3.15 Using a disposable plastic trowel or decontaminated stainless-steel trowel, thoroughly mix (homogenize) the sample material (which now contains a 1-foot interval of sample) in the mixing bowl and fill the appropriate sample bottle(s).

3.16 Fill out a Soil Sample Log Sheet (found in Appendix B, SOP CTO 56-4) and sample labels and tags (according to SOPs CTO 56-5 and CTO 56-10) making sure that the appropriate fields are filled out completely and legibly and affix them to the sample bottle.

3.17 Proceed with handling each sample container as outlined in SOPs CTO 56-2 and CTO 56-3.

3.18 Repeat step steps 3.2 through 3.16 for each 1-foot interval until one of the following conditions have been met. If one of these conditions holds true, the borehole is complete and abandonment procedures outlined in Section 4.10 of the QAPP can begin.

1) The planned depth below ground surface as described in Table 4-1 of the QAPP has been reached;
2) The saturated zone is encountered;
3) Bedrock or weathered bedrock is encountered; or
4) Advancement refusal is met by the hand auger.

3.19 Excess soil from hand augering operations produced during soil sampling will be returned to the bore hole to the extent possible with the remainder to be placed close to where it was collected and raked into the surface as described in Section 4.10 of the QAPP.

3.20 Decontaminate any sampling equipment as described in SOP CTO 56-9.

3.21 Repeat steps 3.4 through 3.17 for each sample location until all samples have been taken.

4.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A GEOPROBE®

At locations where the required penetration can not be reached using the hand auger, Direct Push Technology (DPT) will be employed to collect the sample. DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. It is assumed that this method of sample collection will be required only at a limited number of locations where the required sample depths can not be reached manually using a hand auger.

Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples.

4.1 Drive macrocore samplers (lined with acetate) fitted with detachable 4-foot steel drive points into the ground using hydraulic pressure.

4.2 Retract the sampler from the borehole and remove the 4-foot sample from the hole.

4.3 Attach the metal trough from the Geoprobe ® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

4.4 Place the acetate liner containing the soils in the trough.

4.5 While wearing cut-resistant gloves (constructed of non-latex over cotton), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

4.6 Transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval.

4.7 Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout. All holes should be finished smooth to existing grade.

4.8 Sampling equipment is decontaminated as per SOP CTO 56-9 prior to collecting the next sample.

STANDARD OPERATING PROCEDURE
NUMBER CTO 56-2

SAMPLE PRESERVATION, PACKING, AND SHIPPING
FOR SOIL

STANDARD OPERATING PROCEDURE
NUMBER CTO56-2

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING FOR SOIL

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for sample preservation, packaging, and shipping to be used in handling soil samples obtained for chemical analysis for the Phase 3 Soil RFI for SWMU 7, NSWC Crane.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing Utensil
Shipping Labels: A copy of this label is included in Appendix B, SOP CTO 56-4.
Custody Seals: A copy of this seal is included in Appendix B, SOP CTO 56-4.
Chain-of-Custody (COC) Forms: A copy of this form is included in Appendix B, SOP CTO 56-4.
Sample Containers with Preservatives: All sample containers for analysis by fixed-base laboratories will be supplied and deemed certified clean by the laboratory.
Sample Shipping Containers (Coolers): All sample shipping containers are supplied by the laboratory.
Packaging Material: Bubble wrap, ZipLoc bags®, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

3.1 Table 4-3 of the QAPP establishes requirements for sample preservation. The laboratory provides sample containers that are certified clean for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4°C ±2°C. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.

3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.

3.3 Check that the sample container is properly identified on the label and tag, the lid securely fastened, and the container sealed in a ZipLoc bag.

3.4 Place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut. If the cooler is questionable for shipping, the cooler must be discarded.

3.6 Place the sample container into a shipping cooler in an upright position (containers will be upright). Continue filling the cooler with samples and packing material until the cooler is full and the movement of the sample containers is limited.

3.7 Place a temperature blank in the cooler. Record the temperature of the temperature blank on the COC.

3.8 Fill the voids in between the bubble-out shipping bags with ice and continue filling the cooler with ice to the top, using a minimum of eight pounds of ice for a medium-size cooler.

3.9 Complete a Chain-of-Custody Form (COC) for each cooler. List on the COC the identity each sample bottle contained in the cooler. Include the air bill number on the COC. Use a ballpoint pen and make sure that all of the carbon forms are legible. SOP CTO 56-4 contains instructions for completing the COC. An example of this form can be found in Appendix B, SOP CTO 56-4.

3.10 Place the original (top) signed copy of the COC form, listing only those samples contained in that particular cooler, inside a large ZipLoc bag. Tape the bag to the inside of the lid of the shipping cooler.

3.11 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed, numbered, and dated custody seal to each side of the cooler (a total of four signed custody seals must be used per cooler). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment. SOP CTO 56-4 contains instructions for completing the custody seal and an example of this seal.

3.12 Affix a shipping label to the top of the cooler containing all of the shipping information. Overnight (e.g. FedEx Priority Overnight) courier services will be used for all sample shipments. Include the air bill number on the COC.

3.13 All samples will be shipped to the laboratory no more than 24 hours after completion of sampling. Under no circumstances will sample holding times be exceeded (See Table 4-3 of the QAPP).

STANDARD OPERATING PROCEDURE
NUMBER CTO 56-3

BOREHOLE AND SOIL SAMPLE LOGGING

STANDARD OPERATING PROCEDURE
NUMBER CTO 56-3

BOREHOLE AND SOIL SAMPLE LOGGING

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the standard procedures and technical guidance on borehole and soil sample logging for the Phase 3 Soil RFI for SWMU 7, NSWC Crane.

2.0 FIELD FORMS AND EQUIPMENT

Knife
Ruler (marked in tenths and hundredths of feet)
Boring Log: An example of this form can be found in Appendix B, SOP CTO 56-4.
Writing Utensil

3.0 RESPONSIBILITIES

A field geologist/engineer is responsible for supervising all boring activities and assuring that each borehole is properly and completely logged.

4.0 PROCEDURES FOR BOREHOLE AND SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method classification is detailed in Figure 1 (attached to this SOP).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no distinguishable size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into categories: rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

4.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

4.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in the following table.

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample shall be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

4.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

4.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire field activity.

4.6 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Other distinguishing features

4.7 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table shall be used.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Clay	CL	inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays,
	CH	inorganic clays of high plasticity, fat clays
	OH	organic clays of medium to high plasticity, organic silts
Silt	ML	inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity
	OL	organic silts and organic silty clays of low plasticity
	MH	inorganic silts, micaceous or diatomaceous fine sand or silty soils
Sand	SW	well graded sands, gravelly sands, little or no fines
	SP	poorly graded sands, gravelly sands, little or no fines
	SM	silty sands, sand-silt mixtures
	SC	clayey sands, sand-clay mixtures

FIGURE 1

UNIFIED SOIL CLASSIFICATION (USCS)

FIELD IDENTIFICATION PROCEDURES

(Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)

COARSE-GRAINED SOILS
More Than Half of Material is LARGER Than No. 200 Sieve Sizes

			GROUP SYMBOL	TYPICAL NAMES
GRAVELS (50%(+)>1/4"∅	CLEAN GRAVELS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes	GW	Well graded gravels, gravel sand mixtures, little or no fines
		Predominantly one size or a range of sizes with some intermediate sizes missing	GP	Poorly graded gravels, gravel sand mixtures, little or no fines
	GRAVELS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel sand silt mixtures
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel sand-clay mixtures
SANDS 50%(+)<1/4"∅	CLEAN SANDS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes	SW	Well graded sand, gravelly sands, little or no fines
		Predominantly one size or a range of sizes with some intermediate sizes missing	SP	Poorly graded sands, gravelly sands, little or no fines
	SANDS W/FINES (High % Fines)	Non-plastic fines (for identification procedures see MCL)	SM	Silty sands, poorly graded sand silt mixtures
		Plastic fines (for identification procedures see CL)	SC	Clayey sands, poorly graded sand clay mixtures

FINE-GRAINED SOILS
More Than Half of Material is SMALLER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES
(Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)

Identification Procedures on Fraction Smaller than No. 40 Sieve Size

	DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)	GROUP SYMBOL	TYPICAL NAMES
SILTS AND CLAYS Liquid Limit <50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity
	Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
	Slight to Medium	Slow	Slight	OL	Organic silts and organic silt clays of low plasticity
SILTS AND CLAYS Liquid Limit >50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
	High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays
	Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity
HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture			Pt	Peat and other organic soils

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW GC, well graded gravel sand mixture with clay binder
All sieve sizes on this chart are U.S. Standard

DENSITY OF GRANULAR SOILS	
DESIGNATION	STANDARD PENETRATION RESISTANCE BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS			
CONSISTENCY	UNC COMPRESSIVE STRENGTH (TONS/SQ FT)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail

STANDARD OPERATING PROCEDURE
NUMBER CTO 56-4

SAMPLE CUSTODY AND
DOCUMENTATION OF FIELD ACTIVITIES

STANDARD OPERATING PROCEDURE
NUMBER CTO 56-4

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish the procedures for sample custody and documentation of field sampling and field analyses activities for the Phase 3 Soil RFI for SWMU 7, NSWC Crane. Examples of field forms to be used in this project are provided in Appendix B, attached to this SOP.

2.0 FIELD FORMS LIST

The following log books, forms, labels, and tags are required. Examples of these forms can be found at the back of this SOP.

Writing Utensil
Site Log Book
Field Log Book
Sample Label and Tag
Chain-of-Custody
Custody Seal
Shipping Label
Equipment Calibration Log Sheet
Boring Log
Soil Sample Log Sheet
Ground Water Sample Log Sheet
Monitoring Well Inspection Sheet
Ground Water Level Measurement Sheet
Monitoring Well Development Record
Low-flow Purge Data Sheet
Field Task Modification Request Form

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the log books, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, initialed, and dated.

3.1 Site Log Book

The Site Log Book is a hardbound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded (daily) in the Site Log Book:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues
- Weather conditions

The Site Log Book is initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that onsite activities take place.

The following information must be recorded on the cover of each Site Log Book:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the Site Log Book need not be duplicated in other field notebooks, but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the Site Log Book must be signed and dated by the Field Operations Leader (FOL).

Upon completion of the fieldwork or when completely filled, the Site Log Book is stored in the NSWC Crane records repository.

3.2 Field Log Books

The Field Log Book is a separate dedicated notebook used by field personnel, as needed, to document the activities in the field. This notebook is hardbound and paginated.

Upon completion of the fieldwork or when completely filled, Field Log Books are stored in the NSWC Crane records repository.

3.3 Sample Label and Tag

Adhesive sample container labels must be completed and applied to every sample container. Each adhesive label is numbered. A second, identical (including number) adhesive sample label will be completed and affixed onto a tag that will be attached to the neck of the sample container by a wire or string. Once the laboratory receives the sample, the tag will be removed from the sample container and returned to the Task Order Manager. Sample tags will be stored in the NSWC Crane records repository.

3.4 Chain-of-Custody Form

The Chain-of-Custody (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. Each COC is numbered. This form must be used for any samples collected for laboratory chemical analysis. The original (top) signed copy of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent, a separate COC must be included with each cooler and reflect the sample containers in that particular cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler against the enclosed COC. Any problems are noted on the enclosed COC form (discrepancies between the sample labels, tags, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Task Order Manager. The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

The number of each COC associated with a monitoring point is recorded in the Site Log Book. Each COC is placed into a binder and stored in the NSWC Crane records repository.

3.5 Custody Seal

The Custody Seal is an adhesive-backed label with a number on each seal. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The Custody Seals are signed and dated by the samplers and affixed across the opening edges of each cooler (four seals per medium to larger coolers; two seals per small cooler) containing environmental samples. The laboratory sample custodian will examine the Custody Seal for evidence of tampering and will notify the Task Order Manager if evidence of tampering is observed. The number of each custody seal is recorded on the COC.

3.6 Shipping Label

A shipping label is filled out and attached to goods or samples leaving the site. Most items are shipped via overnight (express) delivery.

3.7 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment (e.g. multi-parameter water quality meter) used in the field. All Equipment Calibration Logs are numbered. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

The number of each Equipment Calibration Log associated with a monitoring point is recorded in the Site Log Book. Each calibration log is placed into a binder and stored in the NSWC Crane records repository.

3.8 Boring Log Sheet

The Boring Log Sheet is used to record the lithology encountered during advancement of the boring. This sheet is used in conjunction with the borehole advancement procedures outlined SOP CTO 56-1 and the lithologic documentation process outlined in SOP CTO 56-3.

3.9 Soil Sample Log Sheet

The Soil Sample Log Sheet is used to document the samples taken from each boring. This sheet is used in conjunction with SOP CTO 56-1 and SOP CTO 56-3.

3.10 Ground Water Sample Log Sheet

The Ground Water Sample Log Sheet is used to document the water samples collected from each well. This sheet is used in conjunction with SOP CTO 56-8.

3.11 Monitoring Well Inspection Sheet

The Monitoring Well Inspection Sheet is used to document the condition of an existing ground water monitoring well.

3.12 Ground Water Level Measurement Sheet

The Ground Water Level Measurement Sheet is used to document the depth to the surface of the ground water within a monitoring well.

3.13 Monitoring Well Development Record

This form is used to record the activities conducted during well development.

3.14 Low-flow Purge Data Sheet

This form is used during the performance of well purging using low-flow techniques.

3.15 Field Task Modification Request Form

This form is used to record any changes from the approved planning documents. Such changes are conducted only after appropriate approval of the TOM and usually in conjunction with prior approval of either the Base, Southdiv or the regulatory agencies.

SAMPLE LABEL AND TAG

Example Sample Label

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Location:** NSWC CRANE		
Sample No:			Tag # :
Date:	Time:		Preserve:
Analysis:			Matrix:
Sampled By:			Laboratory:
Grain Size:	Clay	Silt	Sand

Example Sample Tag

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Location:** NSWC CRANE		
Sample No: :			Tag # :
Date:	Time:		Preserve:
Analysis:			Matrix:
Sampled By:			Laboratory:
Grain Size:	Clay	Silt	Sand

CHAIN-OF-CUSTODY

TETRA TECH NUS, INC.

CHAIN OF CUSTODY | NUMBER | PAGE ____ OF ____

PROJECT NO:	SITE NAME:	PROJECT MANAGER AND PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER AND PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER	CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE PLASTIC (P) or GLASS (G)

PRESERVATIVE USED

TYPE OF ANALYSIS

DATE YEAR	TIME	SAMPLE ID	MATRIX	GRAB (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

YELLOW (FIELD COPY) PINK (FILE COPY) 3/99

CUSTODY SEAL

EXAMPLE OF CUSTODY SEALS

EXAMPLE SHIPPING LABEL

EXAMPLE SHIPPING LABEL

FedEx *USA Airbill* FedEx Tracking Number 8169 3433 4880

Sender's Copy

1 From *Please print and press hard.*

Date

Sender's FedEx Account Number 0152-0168-1

Sender's Name

Phone (412) 821-7090

Company TETRA TECH NUS INC

Address 661 ANDERSEN DR STE 5

Dept./Floor/Suite/Room

City PITTSBURGH State PA ZIP 15220

2 Your Internal Billing Reference

First 24 characters will appear on invoice.

3 To

Recipient's Name

Phone ( )

Company

Address

We cannot deliver to P.O. boxes or P.O. ZIP codes.

Dept./Floor/Suite/Room

To "HOLD" at FedEx location, print FedEx address here.

City State ZIP

NEW Peel and Stick FedEx USA Airbill

See back for application instructions.

Questions? Call 1·800·Go·FedEx® (800-463-3339)

Visit our Web site at www.fedex.com

By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.

0126752955

4a Express Package Service *Packages up to 150 lbs.*

Delivery commitment may be later in some areas.

- [ ] FedEx Priority Overnight — Next business morning
- [ ] FedEx Standard Overnight — Next business afternoon
- [ ] FedEx First Overnight — Earliest next business morning delivery to select locations
- [ ] FedEx 2Day* — Second business day
- [ ] FedEx Express Saver* — Third business day

* FedEx Letter Rate not available. Minimum charge: One-pound rate

4b Express Freight Service *Packages over 150 lbs.*

Delivery commitment may be later in some areas.

- [ ] FedEx 1Day Freight* — Next business day
- [ ] FedEx 2Day Freight — Second business day
- [ ] FedEx 3Day Freight — Third business day

* Call for Confirmation:

* Declared value limit $500

5 Packaging

- [ ] FedEx Letter*
- [ ] FedEx Pak*
- [ ] Other Pkg. — Includes FedEx Box, FedEx Tube, and customer pkg.

6 Special Handling

- [ ] Saturday Delivery — Available for FedEx Priority Overnight and FedEx 2Day to select ZIP codes
- [ ] Sunday Delivery — Available for FedEx Priority Overnight to select ZIP codes
- [ ] HOLD Weekday at FedEx Location — Not available with FedEx First Overnight
- [ ] HOLD Saturday at FedEx Location — Available for FedEx Priority Overnight and FedEx 2Day to select locations

Does this shipment contain dangerous goods?

One box must be checked.

- [ ] No
- [ ] Yes — As per attached Shipper's Declaration
- [ ] Yes — Shipper's Declaration not required
- [ ] Dry Ice — Dry Ice, 9, UN 1845 ____ x ____ kg
- [ ] Cargo Aircraft Only

Dangerous Goods cannot be shipped in FedEx packaging.

7 Payment *Bill to:*

Enter FedEx Acct. No. or Credit Card No. below.

- [ ] Sender — Acct. No. in Section 1 will be billed.
- [ ] Recipient
- [ ] Third Party
- [ ] Credit Card
- [ ] Cash/Check

FedEx Acct. No. / Credit Card No.

Exp. Date

Total Packages	Total Weight	Total Declared Value†	FedEx Use Only
		$ .00	

†Our liability is limited to $100 unless you declare a higher value. See back for details.

8 Release Signature *Sign to authorize delivery without obtaining signature.*

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

359

SRS 989•Rev. Date 11/98•Part #154813S•©1994–98 FedEx•PRINTED IN U.S.A.

EQUIPMENT CALIBRATION LOG SHEET

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ____________________

SITE NAME: ____________________

PROJECT No.: ____________________

INSTRUMENT NAME/MODEL: ____________________

MANUFACTURER: ____________________

SERIAL NUMBER: ____________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: ____________ BORING No.: ____________
PROJECT NUMBER: ____________ DATE: ____________
DRILLING COMPANY: ____________ GEOLOGIST: ____________
DRILLING RIG: ____________ DRILLER: ____________

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION			USCS *	Remarks	PID/FID Reading (ppm)			
					Soil Density/ Consistency or Rock Hardness	Color	Material Classification			Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): [ ]

Remarks: ____

Converted to Well: Yes ________ No ________ Well I.D. #: ____________

SOIL SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL SAMPLE LOG SHEET

Project Site Name: ______________________

Project No.: ______________________

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: ______________________
[] QA Sample Type: ______________________

Sample ID No.: ______________

Sample Location: ______________

Sampled By: ______________

C.O.C. No.: ______________

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:			
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:				
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

FIELD TASK MODIFICATION REQUEST FORM

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name	CTO & Project Number	Task Mod. Number
Modification To (e.g. Work Plan)	Site/Sample Location	Date

Activity Description: ______________________________

Reason for Change: ______________________________

Recommended Disposition: ______________________________

Field Operations Leader (Signature) ______________ Date ______________

Approved Disposition:

____

Project/Task Order Manager (Signature) ______________ Date ______________

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: ______________________________

SAMPLE LABEL AND TAG

Example Sample Label

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Location:** NSWC CRANE	
Sample No:		Tag # :
Date:	Time:	Preserve:
Analysis:		Matrix:
Sampled By:		Laboratory:
Grain Size: Clay	Silt	Sand

Example Sample Tag

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Location:** NSWC CRANE	
Sample No:		Tag # :
Date:	Time:	Preserve:
Analysis:		Matrix:
Sampled By:		Laboratory:
Grain Size: Clay	Silt	Sand

CHAIN-OF-CUSTODY

TETRA TECH NUS, INC.

CHAIN OF CUSTODY | NUMBER | PAGE ____ OF ______

PROJECT NO:	SITE NAME:	PROJECT MANAGER AND PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER AND PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER	CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE PLASTIC (P) or GLASS (G)

PRESERVATIVE USED

TYPE OF ANALYSIS

DATE YEAR	TIME	SAMPLE ID	MATRIX	GRAB (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

YELLOW (FIELD COPY) PINK (FILE COPY) 3/99

CUSTODY SEAL

EXAMPLE OF CUSTODY SEALS

Signature

Date

CUSTODY SEAL

CUSTODY SEAL

Date

Signature

SEAL #
00001

EXAMPLE SHIPPING LABEL

EXAMPLE SHIPPING LABEL

FedEx *USA Airbill* FedEx Tracking Number 8169 3433 4880

Sender's Copy

1 From *Please print and press hard.*

Date | Sender's FedEx Account Number 0152-0168-1

Sender's Name | Phone (412) 821-7090

Company TETRA TECH NUS INC

Address 661 ANDERSEN DR STE 5

Dept./Floor/Suite/Room

City PITTSBURGH State PA ZIP 15220

2 Your Internal Billing Reference
First 24 characters will appear on invoice.

3 To
Recipient's Name | Phone ( )

Company

Address

We cannot deliver to P.O. boxes or P.O. ZIP codes. | Dept./Floor/Suite/Room

To "HOLD" at FedEx location, print FedEx address here.

City | State | ZIP

NEW Peel and Stick FedEx USA Airbill
See back for application instructions.

Questions? Call 1·800·Go·FedEx® (800-463-3339)
Visit our Web site at www.fedex.com
By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.

0126752955

4a Express Package Service — *Packages up to 150 lbs.* Delivery commitment may be later in some areas.

- ☐ FedEx Priority Overnight — Next business morning
- ☐ FedEx Standard Overnight — Next business afternoon
- ☐ FedEx First Overnight — Earliest next business morning delivery to select locations
- ☐ FedEx 2Day* — Second business day
- ☐ FedEx Express Saver* — Third business day

* FedEx Letter Rate not available. Minimum charge: One-pound rate

4b Express Freight Service — *Packages over 150 lbs.* Delivery commitment may be later in some areas.

- ☐ FedEx 1Day Freight* — Next business day
- ☐ FedEx 2Day Freight — Second business day
- ☐ FedEx 3Day Freight — Third business day

* Call for Confirmation:

5 Packaging — * Declared value limit $500

- ☐ FedEx Letter*
- ☐ FedEx Pak*
- ☐ Other Pkg. — Includes FedEx Box, FedEx Tube, and customer pkg.

6 Special Handling

- ☐ Saturday Delivery — Available for FedEx Priority Overnight and FedEx 2Day to select ZIP codes
- ☐ Sunday Delivery — Available for FedEx Priority Overnight to select ZIP codes
- ☐ HOLD Weekday at FedEx Location — Not available with FedEx First Overnight
- ☐ HOLD Saturday at FedEx Location — Available for FedEx Priority Overnight and FedEx 2Day to select locations

Does this shipment contain dangerous goods? One box must be checked.

- ☐ No
- ☐ Yes — As per attached Shipper's Declaration
- ☐ Yes — Shipper's Declaration not required
- ☐ Dry Ice — Dry Ice, 9, UN 1845 ____ x ____ kg
- ☐ Cargo Aircraft Only

Dangerous Goods cannot be shipped in FedEx packaging.

7 Payment *Bill to:* Enter FedEx Acct. No. or Credit Card No. below.

- ☐ Sender — Acct. No. in Section 1 will be billed.
- ☐ Recipient
- ☐ Third Party
- ☐ Credit Card
- ☐ Cash/Check

FedEx Acct. No. / Credit Card No. | Exp. Date

Total Packages	Total Weight	Total Declared Value†	
		$ .00	FedEx Use Only

†Our liability is limited to $100 unless you declare a higher value. See back for details.

8 Release Signature *Sign to authorize delivery without obtaining signature.*

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

359

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EQUIPMENT CALIBRATION LOG SHEET

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ______________________ INSTRUMENT NAME/MODEL: ______________________

SITE NAME: ______________________ MANUFACTURER: ______________________

PROJECT No.: ______________________ SERIAL NUMBER: ______________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: ______________ BORING No.: ______________
PROJECT NUMBER: ______________ DATE: ______________
DRILLING COMPANY: ______________ GEOLOGIST: ______________
DRILLING RIG: ______________ DRILLER: ______________

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	U S C S *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: ______________________________

Converted to Well: Yes ______ No ______ Well I.D. #: ______________

SOIL SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL SAMPLE LOG SHEET

Page___ of___

Project Site Name: ______________________

Project No.: ______________________

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: ______________________
[] QA Sample Type: ______________________

Sample ID No.: ______________________

Sample Location: ______________________

Sampled By: ______________________

C.O.C. No.: ______________________

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:			
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:				
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

FIELD TASK MODIFICATION REQUEST FORM

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name	CTO & Project Number	Task Mod. Number
Modification To (e.g. Work Plan)	Site/Sample Location	Date

Activity Description: ______

Reason for Change: ______

Recommended Disposition: ______

Field Operations Leader (Signature) ______ Date ______

Approved Disposition: ______

Project/Task Order Manager (Signature) ______ Date ______

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: ______

STANDARD OPERATING PROCEDURE

NUMBER CTO 56-5

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

STANDARD OPERATING PROCEDURE

NUMBER CTO 56-5

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish the procedures to be followed when decontaminating non-dedicated field sampling equipment for the Phase 3 Soil RFI for SWMU 7, NSWC Crane

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing Utensil
Non-latex Gloves
Cotton Gloves
Field Log Book
Potable Water
Deionized Water
LiquiNox Detergent
Brushes, Spray Bottles, Paper Towels, etc.

3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the Field Log Book.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in 4.10 of the QAPP.

STANDARD OPERATING PROCEDURE

NUMBER CTO 56-6

SAMPLE IDENTIFICATION NOMENCLATURE

STANDARD OPERATING PROCEDURE

NUMBER CTO56-6

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management for the SWMU 7 Phase 3 Soils RFI QAPP, NSWC Crane. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Maintenance of consistency (field, laboratory, and data base sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of identification and direct link to site and year.

The NSWC Crane Environmental Protection Department must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with Indelible Ink
Sample Tags
Sample Container Labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Environmental Samples

All environmental samples taken as part of this QAPP at NSWC Crane will be properly labeled with a sample label affixed to the sample container and a sample tag tied around the neck of the sample container. Each sample will be assigned a unique sample tracking number. The sample tracking number will consist of a four segment alpha-numeric code that identifies the sample's associated solid waste management unit (SWMU) or associated site, sample type, and location. For soil samples, the final four tracking numbers will identify the depth at which the soil or sediment sample was collected.

The alpha-numeric coding to be used in the NSWC Crane sample system is explained in the diagram and the subsequent definitions:

Soil Samples:

NN	AA	AA	(NNNN)
SWMU Number	Sample Type	Location	Depth Interval

Character Type:

A = Alpha
N = Numeric

SWMU Number:

07 = Old Rifle Range

Sample Type:

CP = Composite Soil Sample
SS = Surface Soil Sample
SB = Subsurface Soil Boring Sample

Location:

The sample location code is the soil sample location. The location code for each sample is listed on figures and tables in the Site-specific Work Plan.

Location 1 = 01
Location 2 = 02, etc

Depth Interval:

The depth code is used to note the depth below ground surface (bgs), at which a soil sample is collected. The first two numbers of the four number code specify the top interval and the third and fourth specify the bottom, feet bgs of the sample. The depths will be noted in whole numbers only, further detail, if needed, will be recorded on the sample log sheet, boring log, log book, etc.

0001 = soil collected from 0 to 1 foot bgs
0204 = soil collected from 2 to 4 feet bgs

For example, a surface soil sample collected from 0 to 1 foot at sampling location 04 in the ORR will be designated as 07SS040001, or a subsurface soil sample collected from 3 to 4 feet at sampling location 05 in the ORR will be designated as 07SB050304. Note: there is no differentiation between samples collected at the Old Rifle Range (ORR) and the Old Pistol Range (OPR); samples collected from either location will be identified as 07 (indicating SWMU 7).

4.0 FIELD QUALITY ASSURANCE/ QUALITY CONTROL (QA/QC) SAMPLES

Field QA/QC samples are described in Section 8.1 of the QAPP. They will be designated with a different coding system. The QC code will consist of a three-segment, alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC samples collected on that date. The QC types are identified as:

AA	**NNNNNN**	**NN**
QC Type	Date	Sequence Number (per day)

The QC types are identified as:

- SW = Source Water Blank
- RB = Rinsate Blank (Equipment Blank)
- FB = Field Blank
- FD = Field Duplicate
- TB = Trip Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

Examples of Field QA/QC Sample Nomenclature

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

PHOTOIONIZATION DETECTOR

User's Manual

2020

PHOTOIONIZATION AIR MONITOR

PHOTOVAC

Registered to the ISO 9002 International Quality Standard

Warning: Limitation of Liability

The ultimate responsibility of the consequences of use of toxic compounds rests with the user. Photovac's role is as a supplier of instrumentation to assist in the early detection of hazardous conditions involving such compounds.

2020 represents a major advance in this field and, as with all complex instruments, it is vitally important to ensure that 2020 is maintained in accordance with Photovac's instructions and that proper calibration is regularly performed.

As with any complex device, 2020 is subject to failure and, while Photovac has taken, and continues to take, all possible precautions to (a) reduce the possibility of failure, and (b) warn the user in the event of failure, circumstances may occasionally occur in which there is a failure despite such precautions on Photovac's part. Photovac regrets that it cannot accept liability for damages of any kind caused as a result of either failure of the user to follow instructions or of 2020 to perform.

2020

Photoionization Air Monitor

User's Manual

Photovac Incorporated
330 Cochrane Drive
Markham, Ontario
L3R 8E5, Canada
Tel (905)477-8088
Fax (905)477-8220

Photovac Monitoring Instruments
25-B Jefryn Boulevard West
Deer Park, New York
11729, USA
Tel ~~(516)254-4199~~
Fax (516)254-4284
(203) 761 - 5330

Photovac Europa A/S
Sondervang 19
DK 4100 Ringsted
Denmark
Tel +45-5767-5008
Fax +45-5767-5018

Part No. 350001 Rev

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Trademarks

Reference No. 600762

Table of Contents

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Notices and Warnings

FCC Warning

This equipment has been tested and found to comply with the limits for a Class B Digital Device, pursuant to Subpart B, Class B of Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

2020 I/S Notice

Listed
77463

CONFORMS TO
UL STANDARD 913

CERTIFIED TO
CSA STANDARD
CSA 22.2 No. 157

Intrinsically safe/Sécurité intrinsèque for use in Class I, Division 1, Groups A, B, C, D, Hazardous Locations, Temperature Code T4, Exia

2020 I/S IS CLASSIFIED FOR USE IN CLASS I, DIVISION 1, GROUPS A, B, C, D HAZARDOUS LOCATIONS. T4 (135°C) RATING.

It has been listed by ETL® to comply with Underwriters Laboratories® Inc. UL® 913 *Standard for Intrinsically Safe Apparatus and Associated Apparatus for use in Class I, Division I, Groups A, B, C, D Hazardous (Classified) Locations*, Fourth Edition.

2020 I/S IS NOT INTENDED TO DETECT COMBUSTIBLE LEVELS OF GASES. 2020 I/S IS CLASSIFIED FOR USE IN ATMOSPHERES CONTAINING COMBUSTIBLE LEVELS OF GASES.

These Photovac accessories are for use with 2020 I/S in a hazardous location:

350006	Calibration Regulator
350007	Wrist Strap
350008	Belt-Clip Holster
350010	Carrying Case
350014	User's Reference Card
390006	Long Sample Probe
395001	Short Sample Probe

Do not use any other accessories with 2020 I/S in a hazardous location.

Substitution of components may affect safety rating.

CAUTION

To reduce the risk of fire or injury to persons, read and follow these instructions:

1. All calibration, maintenance and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.
2. For replacement battery pack use only Photovac Part No. 350009.
3. Do not dispose of the battery pack in a fire. The cell may explode. The battery pack must be disposed of properly or returned to Photovac for recycling. Check with local codes for possible special disposal instructions.
4. Do not open or mutilate the battery pack.
5. Exercise care in handling battery packs in order not to short the terminals with conducting materials such as rings, bracelets and keys. The battery or conductor may overheat and cause burns.
6. Do not defeat proper polarity orientation between the battery pack and battery charger.

7. Charge the battery pack using the AC adapter provided with or identified for use with this product only in accordance with the instructions and limitations specified in this manual. For AC adapter use only Photovac Part No. 350002 (115 Volts AC), 396013 (220 Volts AC).

ATTENTION

2020 I/SC EST CLASSIFIÉ POUR USAGE DANS LES EMPLACEMENTS DANGEREUX DE CLASSE I, DIVISION 1, GROUPES A, B, C, D. ÉVALUATION T4 (135°C).

2020 I/SC est conforme à la norme des Underwriters Laboratories Inc. UL 913 *Standard for Intrinsically Safe Apparatus and Associated Apparatus for use in Class I, Division 1, Groups A, B, C, D Hazardous (Classified) Locations*. Quatrième édition.

2020 I/SC est conforme à la norme de *CSA Standard 22.2 No. 157-92 - Intrinsically Safe and Non-Incendive Equipment for Use in Hazardous Locations*.

2020 I/SC C'EST NE PAS INTENDER POUR DÉTECTER DES NIVEAUX DE COMBUSTION DES GAZ. CET APPAREIL EST CLASSIFIÉ POUR USAGE DANS DES ATMOSPHÈRES CONTENANT DES NIVEAUX DE COMBUSTION DES GAZ.

Les accessoires Photovac suivants peuvent également être utilisés avec l'appareil dans un emplacement dangereux:

350006	Régulateur de calibration
350007	Sangle de poignet
350008	Étui de ceinture
350010	Étui de transport
350014	Carte de référence
395001	Petite Gamme d'échantillons
396018	Gamme d'échantillons

Ne pas utiliser d'autres accessoires avec cet appareil dans un emplacement dangereux.

La substitution des composantes peut nuire à la sécurité d'emploi.

MISE EN GARDE

Afin de réduire les risques d'incendie et les blessures, lire et suivre ces instructions:

1. Tout étalonnage, entretien et réparation de cet appareil, y compris le chargement de la pile, doit être effectué dans un endroit sûr, à l'écart des zones dangereuses.
2. N'utiliser que la pièce Photovac numéro 350009 lorsqu'il faut remplacer le bloc-pile.
3. Ne pas jeter la pile dans un feu. La cellule pourrait exploser. Vérifier les codes locaux, qui peuvent comporter des instructions de mise au rebut particulières.
4. Ne pas ouvrir et ne pas abîmer le bloc-pile.
5. Manipuler le bloc-pile avec soin, afin de ne pas court-circuiter les bornes avec des matériels conducteurs tels qu'une bague, un bracelet ou des clés. La pile ou le conducteur pourraient surchauffer et causer des brûlures.
6. Ne pas modifier l'orientation de la polarité appropriée entre le bloc-pile et le chargeur.
7. Charger le bloc-pile fourni avec ou reconnu pour usage avec ce produit seulement conformément aux instructions et restrictions spécifiées dans ce manuel. Pour le chargeur, n'utiliser que la pièce Photovac numéro 350002 (115 Volts AC), 396013 (220 Volts AC).

1. Introduction

1.1. About this Manual

This manual provides detailed instructions for setup, operation and maintenance of the Photovac 2020™ Photoionization Air Monitor.

Before unpacking the instrument, please read Section 1.2 Warnings and Safety Practices. This section describes possible hazards that might injure the user, damage the instrument or compromise its operation. Some general safety information is also provided.

To help you learn to use 2020 quickly, this manual is organized by tasks beginning with a tutorial and description of operation in Chapter 2. More detailed operational instructions are provided in Chapter 3. Accessories is covered in Chapter 4. Routine maintenance is covered in Chapter 5. Troubleshooting techniques are covered in Chapter 6. Chapter 7 provides a technical description of 2020.

The 2020 manual uses a few conventions for key names on the keypad and for text that is shown on the display.

UPPERCASE	Fixed key names are denoted by uppercase text.
"Display Text"	Text that appears on the 2020 status display is in quotation marks.
	Soft key names are also shown in quotation marks.

<Angle Brackets>	Computer keyboard names are denoted by angle brackets, e.g. <Ctrl>.
C:\2020	Text that must be typed in using the computer keyboard is shown in italics.

In the text you will find various warnings and notes.

Warning: A warning indicates an operation that could cause personal injury if precautions are not followed.

Note: A note indicates an operation that could cause instrument damage if precautions are not followed. A note also indicates significant information.

1.2. Warnings and Safety Practices

Please read this section before operating 2020.

1.2.1. Approved Model of 2020

This manual provides operational information for all models of 2020. The 2020 I/S, is intrinsically safe and approved for use in hazardous locations. Refer to the manual introduction for details of the approval.

Throughout the manual, notes are provided to inform you of the limitations of usage for the 2020 I/S model.

Warning: If the 2020 model you are using is not specifically identified as intrinsically safe, do not use it in a location were flammable concentrations of gases and vapors may exist.

1.2.2. Compressed Gases

Cylinders of compressed gas, such as calibration gas, must be handled with extreme care. For safety, the calibration gas cylinders must be secured before use.

Please observe the following handling procedures:

1. Mark each new regulator with its intended gas service and never use a regulator for more than one service. To ensure safety and avoid contamination, regulators should be dedicated to one service. Do not change gas service or adapt equipment without consulting your gas supplier.
2. Do not heat or expose cylinders or regulators to temperatures above 52°C (125°F). The cylinders may rupture at high temperatures.
3. Use only the specified regulator for the calibration gas. Confirm regulator type and material with your specialty gas supplier.
4. Always secure cylinders before removing the cylinder valve protection cap.
5. Do not drag or roll cylinders. Large cylinders should only be moved on carts designed for compressed gas cylinders. Do not move cylinders without the valve protection cap in place.
6. Wear safety glasses when working with compressed gases.
7. Never use the regulator as a shut-off valve. Close the cylinder when it is not in use.
8. Do not store cylinders in a hazardous location. Store cylinders in an upright position away from possible sources of heat or sparks.
9. Do not move or detach the regulator when it is pressurized or when it is in use.
10. Do not subject the regulator to an inlet pressure greater than recommended.
11. Never plug, obstruct or tamper with safety relief devices.
12. Before connection, ensure the gas cylinder valve and the regulator CGA connection are clean.

1.2.3. Regulators for Compressed Gases

When connecting a regulator to a large cylinder:

1. Ensure cylinder valve and regulator connection match.
2. Ensure regulator construction materials are compatible with the gas, and that the cylinder pressure gauge will withstand the cylinder pressure.

3. Turn the pressure control valve on the cylinder all the way out (close the cylinder).
4. Turn the regulator outlet to off.
5. Open the gas cylinder valve slowly and check for leaks.
6. Adjust the delivery pressure.
7. Open the regulator outlet valve.

1.2.4. Calibration Gas

Adequate ventilation must be provided when 2020 is being calibrated.

If compound threshold limit values (TLV™) are exceeded, you should use a gas bag for sampling and calibration.

To determine the TLV of the compounds contained in the calibration gas, refer to the Material Safety Data Sheet (MSDS) supplied with your calibration gas cylinder. See Section 3.3 for details of calibration using a gas bag.

1.2.5. Battery Pack Care

Leaving 2020 for more than 3 months, without turning it on, may result in the loss of recorded data and setup parameters. If 2020 is not used for long periods of time, turn the instrument on for a few hours every month to avoid loss of data. See Section 1.5 for instructions on charging the battery.

Please observe the following:

1. For replacement battery pack use only Photovac Part No. 350009.
2. Do not dispose of the battery pack in a fire. The cell may explode. Check with local codes for possible special disposal instructions. This battery pack must be disposed of properly or returned for recycling to the nearest Photovac facility.
3. Do not open or mutilate the battery pack.
4. Exercise care in handling battery packs in order not to short the terminals with conducting materials such as rings, bracelets and keys. The battery or conductor may overheat and cause burns.

5. Charge the battery pack using the AC adapter provided with or identified for use with this product only in accordance with the instructions and limitations specified in this manual. For AC adapter use only Photovac Part No. 350002 (North America) or 396013 (Europe).

1.2.6. Excessive Heat and Cold

Do not expose the instrument to intense sunlight for prolonged periods.

Exposure to excessive heat or cold may result in erroneous readings.

1.3. Unpacking

The following accessories are included with your 2020:

1. Sample Probe
2. User's Manual
3. Multi-Tool
4. AC Adapter or AC Adapter with AC Line Cord
5. Wrist Strap
6. Replacement Sample Inlet Filters (10 pieces)
7. Reference Card

Ensure all of these accessories have been included with the instrument. If any items are missing or damaged, contact Photovac immediately.

1.4. Support Equipment and Consumables

1.4.1. Calibration

For normal operation these items are required:

1. Calibration Gas Regulator (Photovac Part No. 350006).
2. Calibration gas containing 100 ppm isobutylene. (Photovac Part No. 350012). Other concentrations of the calibration gas may be required. This will depend on your application.
3. Zero air. Occasionally, clean, ambient air is suitable for calibration.

Alternatively, you can use a gas sampling bag and a source of hydrocarbon free air. Air should not have more than 0.1 ppm total hydrocarbons (THC).

If you will be using large tanks of gas, specify a single stage, high purity regulator with a CGA 590 connection at the inlet. The regulator should also have a 1/8" parallel, compression fitting with which to connect the regulator to the gas bag adapter. The delivery pressure must be adjustable to between 5 and 20 psig (34.5 and 138 kPa). You may obtain a gas bag and gas bag adapter (Photovac Part No. 395072 - gas bag, 395073 - gas bag and adapter).

4. If compound threshold limit values (TLVs) are exceeded, you should use a gas bag for sampling and calibration.

   To determine the TLV of the compounds contained in the calibration gas, refer to the Material Safety Data Sheet (MSDS) supplied with your calibration gas cylinder.

   If you will be using a gas bag for calibration, you should obtain the calibration kit (Photovac Part No. 390033). The calibration kit contains a regulator, a gas sampling bag and a gas bag adapter. See Section 3.3 for details of calibration using a gas bag.

1.4.2. Field Operation

For field operation, a 2020 Field Kit (Photovac Part No. 350005) is available. The field kit includes a cable kit, a carrying case and a calibration regulator, spare battery pack and a cylinder of calibration gas.

Refer to the check list in Section 3.7 for additional items. Ensure you have all the necessary accessories and equipment before beginning field operation.

1.4.3. Printer

2020 may be used with a printer to generate printed reports. The printer must have a printing width of at least 65 characters and must use fixed spaced fonts. See Section 4.1 for details of connecting a printer to 2020.

If you are using a parallel printer, you will need the Photovac serial to parallel converter (Photovac Part No. 380145). See Section 4.3

for details of connecting and operating 2020 with a serial to parallel converter.

Note:	2020 I/S is not classified for use in hazardous locations with a printer.

1.4.4. Computer

2020 may also be connected to a computer. The computer must be 100% compatible with an IBM PC. You can use the cable kit (Photovac Part No. 350011) to connect 2020 to the computer.

You will also need terminal emulation software. Software packages such as Crosstalk®, and Procomm® are recommended for use with 2020. If you are using Microsoft® Windows™ you do not need to purchase any separate software. If you are already using another type of communication or terminal emulation software package, it is not necessary to purchase separate software for 2020. See Section 4.2 for details.

Note:	2020 I/S is not classified for use in hazardous locations with a computer.

1.5. Battery Charging

Before beginning operation of 2020, the battery pack must be charged. You can also remove the battery pack and replace it with a fully charged, spare battery pack (Photovac Part No. 350009).

1.5.1. Removing and Replacing the Battery Pack

Note:	Do not remove or recharge the battery pack in a hazardous location

1. If the instrument has been turned on, turn it off by pressing the ON/OFF key momentarily and then releasing it.

Note:	If you do not turn 2020 off before removing the battery pack, you will reset the instrument and you will lose all logged data and setup parameters.

2. Locate the battery hatch on the back of the instrument. See Figure 1.
3. Loosen the two screws in the battery hatch.
4. The battery hatch can now be removed.
5. Lift the battery pack out of the case and carefully disconnect the connector from 2020.
6. Attach the connector from the charged battery pack to the 2020.

Note:	The connector is polarized. It will only fit one way. Do not force the connection.

7. Place the battery pack in the 2020 case. Ensure the battery wires are not pinched or strained.
8. Replace the battery hatch and then replace the two screws. Do not over-tighten the screws as you will damage the case.

1.5.2. Charging the Battery Pack

Note:	Only use the AC adapter specified for use with 2020. Using another AC adapter will result in damage to the battery pack, 2020 or the adapter itself.

1. Plug the AC adapter into the jack located on the back of the 2020. See Figure 1.
2. Plug the AC adapter into an AC outlet. If you are using the European AC adapter, ensure the correct plug is installed on the line cord. If it is not correct for the wall outlet in your area then it must be replaced. See Appendix 8.3.
3. The LED on the 2020 indicates the charge state. Red indicates the battery is being charged. Green indicates the battery is fully charged and ready for use.

   It is normal for a fully charged battery to indicate it is charging (red light) when first plugged in. The LED will turn green as the battery charges.
4. When the battery pack is fully charged remove the AC adapter, first from the wall outlet and then from 2020.

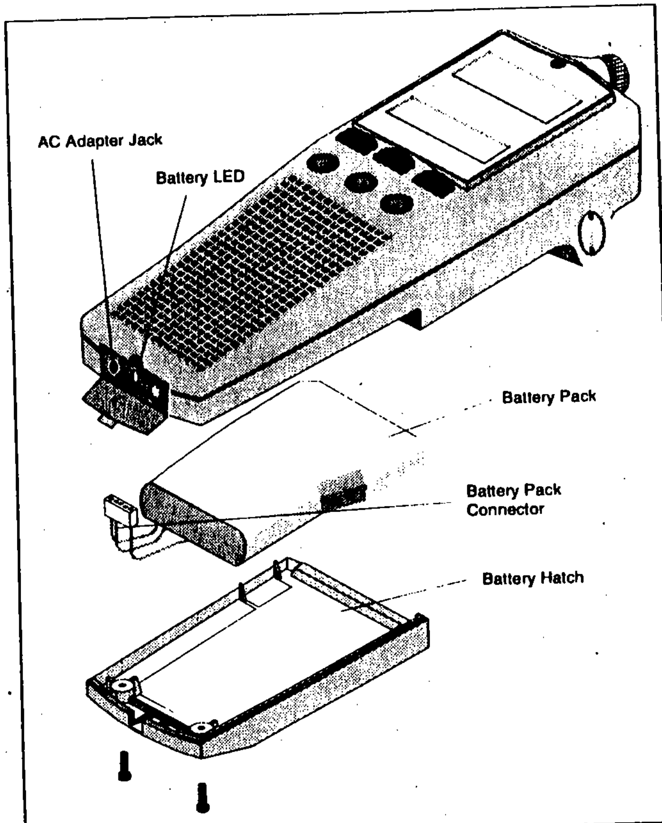

Figure 1 Battery Pack Removal and Replacement

Charging a fully discharged battery pack will take approximately 4 hours.

You must charge the battery pack in the instrument. While the battery pack is charging you can use all the features of 2020.

Leaving the AC adapter connected to 2020 will not harm the battery or the AC adapter in any way. If 2020 is to be left indefinitely, leave it connected to the AC adapter so that the battery will be fully charged and ready for operation.

On average a fully charged battery pack will provide 8 hours of continuous operation. Battery life is shorter if the instrument is turned off and then on again repeatedly or if the backlighting is turned on.

1.6. Overview

2020 measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations. It does not distinguish between individual pollutants. The reading displayed represents the total concentration of all photoionizable chemicals present in the sample. 2020 is factory set to display concentration in units of ppm or mg/m^3.

2020 operates automatically. The meter display updates itself once per second. You can read concentrations directly from the meter display. If you are using the dilution probe (Photovac Part No. 350013) you must multiply the displayed reading by the dilution factor. See Section 4.5 for details.

2020 is always performing short term exposure limit (STEL), time weighted average (TWA) and PEAK calculations. You can view any of these results, but only one mode may be viewed at a time.

2020 has many datalogging options. You can select an averaging interval, or you can use manual operation. If you select an averaging interval, the minimum, maximum, and average concentrations measured in each period are automatically recorded in 2020's datalogging memory. 2020 can log up to 1000 entries

In manual operation, 2020 prompts you to locate a site and then to record a background and sample readings for the site. You can record up to 1000 manual entries. There is no averaging of data in manual operation.

Recorded data can be reviewed on the display, sent to a printer in either tabular or graphical format, or sent to a computer. Data are recorded by date and time.

2020 has 6 keys for alphanumeric entry and for accessing 2020's functions. The keys are used to set up and calibrate 2020. They allow you to manipulate the concentration data in various ways.

All information entered with the keys and stored in 2020's memory is retained when the instrument is switched off. The clock and calendar continue to operate and do not need to be set each time 2020 is turned on.

2. Tutorial Session

2.1. Displays

The 2020 has a meter display for reporting detected concentration, and a status display used to display status information and guide you through configuration options. All functions of the 2020 will be controlled or reported using one of these displays.

2.1.1. Meter Display

The meter display is a 4 digit display. It will always be used for reporting detected concentration. When the detector and pump are off, the meter display will be blank.

In order to accommodate the range of concentrations 2020 can detect, the meter reading will be reported using one of 2 resolutions. A resolution of 0.1 will be used for concentrations below 100 ppm, and a resolution of 1 will be used for concentrations above 100 ppm.

The meter display reports PEAK, MAX, STEL or TWA concentration. The update rate is dependent on the reading being reported. See Section 2.5 for a description of these measurements.

2.1.2. Status Display

The status display is a 2 line by 16 character display. The top line is used to display status information and prompts you for information. The bottom line is used for soft key names. Up to 3 names can be displayed for the 3 soft keys. If a name does not appear for a soft key, then the soft key has no associated function.

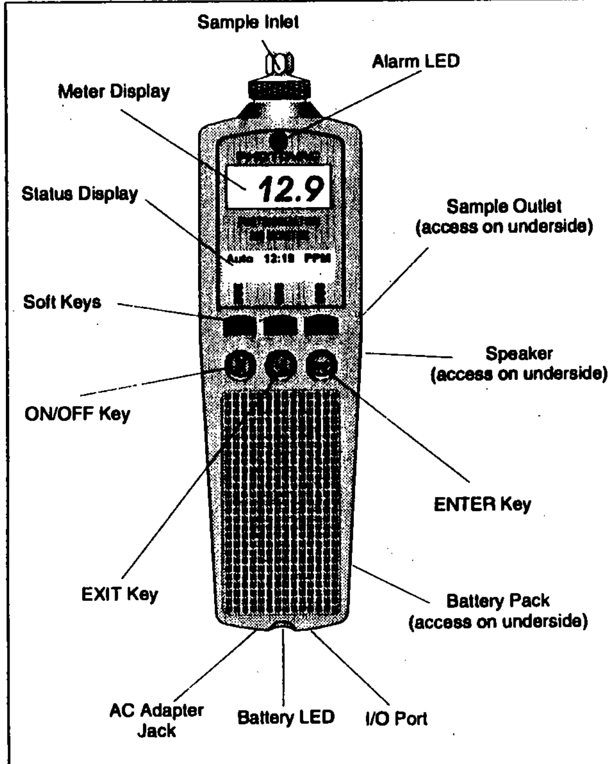

Figure 2 Layout of 2020

2.2. Keys

2.2.1. Fixed Keys

The three round keys below the soft keys each have a fixed function. The first key is the ON/OFF key, the middle key is the EXIT key and the last key is the ENTER key.

The ON/OFF key is used to both turn power on to the 2020 as well as turn the power off. To turn on 2020, press the ON/OFF key. To

turn the power off, press the ON/OFF key and hold it down for 2 seconds, and then release it. This is done to prevent accidental power off.

The EXIT key provides a way of returning to the default display. In the functional map, the soft keys allow you to advance and the EXIT key provides a way to go back. See Figure 3. If you are at the root entry of the menu, EXIT will return you to the default display.

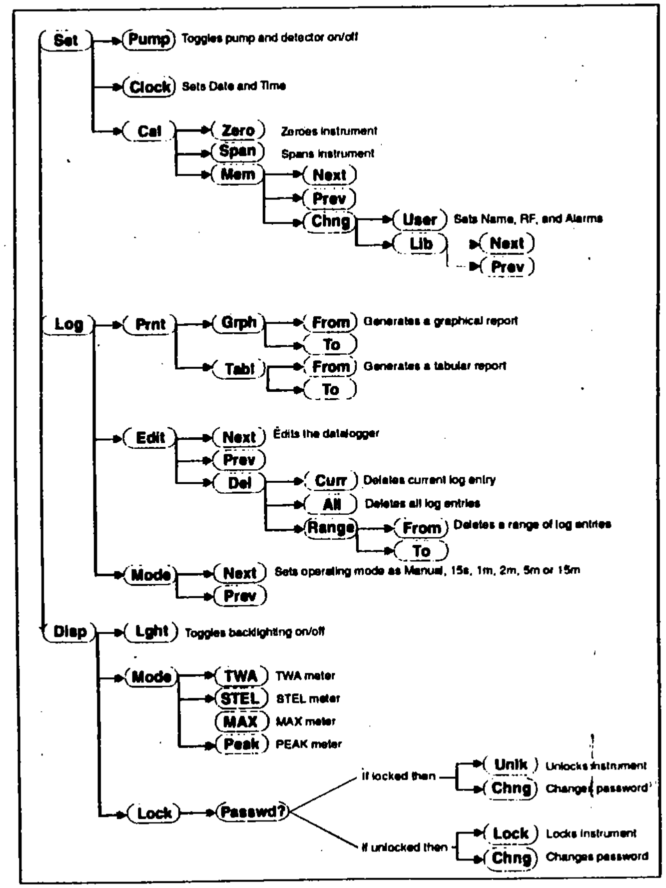

Figure 3 Function Map

The ENTER key has a context sensitive function. When you are operating or navigating through the function map, the ENTER key is used to exit the functions and return you to the default display. When entering data such as a name, number, date or time, ENTER is used to confirm the entry.

2.2.2. Soft Keys

The three soft keys on 2020 are located directly below the status display. Each key has varying functions for configuring 2020, editing the datalogger, and controlling the display. Since only three soft keys are available, each function is broken down into a path. A map, showing each path and the resulting functions, is shown in Figure 3.

2.2.3. Entering Text With the Soft Keys

For all information that you must enter, the left, center and right soft keys correspond to the up, down, and right arrow. See Figure 4.

The up and down arrows are used to change the character highlighted by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right most character, it wraps around to the first character again. To accept the changes, press the ENTER key. To ignore the changes, press EXIT.

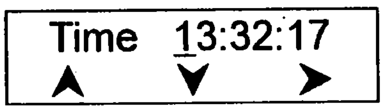

Figure 4 Soft Arrow Keys

Formatting characters, such as the colon (:) in the time, the decimal (.) in a concentration, and the slash (/) in date are skipped when advancing the cursor.

All inputs use an 8 character input, which is displayed on the right side of the top line of the status display. The prompt, describing the input, occupies the left half of the top line. The soft keys are defined on the bottom line of the status display.

2.3. Beginning Operation

2.3.1. Turning 2020 On

1. Turn 2020 on by pressing the ON/OFF key. See Figure 2 for the location of the ON/OFF key.
2. 2020 will display the software version number. Wait for the 2020 to proceed to the default display.
3. Allow 10 minutes for the instrument to warm-up and stabilize.

2.3.2. Default Display

The meter display shows the detected concentration. The resolution of the display changes with the magnitude of the reading. A reading of 0 to 99.9 will be displayed with a resolution of 0.1 ppm or mg/m³. A reading greater than 99.9 will be shown with a resolution of 1 ppm or mg/m³. The meter will display concentrations up to 2000 ppm or 2000 mg/m³.

The status display is used to display the instrument status, date, time, units and active soft keys. Figure 5 shows the status displays.

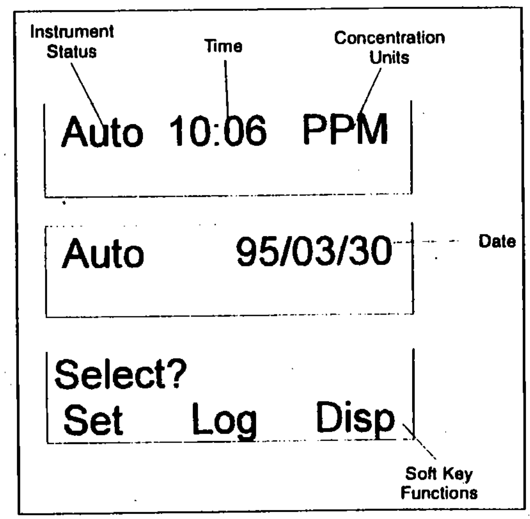

Figure 5 Status Displays

The default display provides the following information; instrument status, current detected concentration, time, date and measurement units. The status display toggles between showing time and units and then the date.

When the display mode is MAX, the date and time correspond to the date and time the MAX concentration was recorded. In TWA mode, the time represents the number of hours and minutes during which the TWA has been accumulating. For PEAK and STEL monitoring, the date and time correspond to the current date and time.

2.4. Monitoring

2.4.1. Instrument Status

The instrument status is shown on the left of the first line of the status display and on the Table and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off. Table 1 is a list of the possible status messages.

Status	Description
Falt1	Zero fault. See Section 6.2.
Falt2	Span fault. See Section 6.2.
Falt3	Lamp fault. See Section 6.2.
Falt4	Pump fault. See Section 6.2.
Cal	Will not be observed on the display during normal operation as various calibration prompt messages are displayed during calibration. If the instrument is turned off during calibration, Cal will be displayed when 2020 is turned on again to indicate the last calibration was incomplete.
Over	Instrument over range. The detector electronics have become saturated. See Section 6.2.
ALPK	Detected concentration exceeds the PEAK alarm level.
ALST	STEL alarm level has been exceeded.
ALTW	TWA alarm level has been exceeded.
LBat	Battery pack power is low.
ELog	The datalogger is full.

Table 1 Instrument Status

Status	Description
TWA	TWA display mode.
MAX	MAX display mode.
STEL	STEL display mode.
Auto	PEAK display mode.
Loc	Locate site. Used for manual operation.
BkGd	Record background reading. Used for manual operation.
Samp	Record sample reading. Used for manual operation.
Off	Pump is off

Table 1 Instrument Status

2.4.2. Alarms

While operating the instrument, any one of three alarm conditions can occur. To accurately identify the source of the alarm, each type of alarm has been given a unique status. See Table 1.

In addition to the status, 2020 also has an audio alarm and an alarm LED. To conserve power the 2020 alternates between the LED and audio. Different alarms are identified by the frequency at which the 2020 alternates between the audio and LED; Peak alarm 5 times per second, STEL alarm 2.5 times per second, and TWA alarm is 1.25 times per second.

The left soft key is used for acknowledging alarms, and is named "Ack". If no alarm exists, then the "Ack" key is not shown. To clear the alarm, press the "Ack" key. Once acknowledged, the alarm indicators are cleared. The alarm status will remain until the alarm condition clears.

2020 updates the peak concentration once every second. Following every update, the peak concentration is compared to the peak alarm level, and if exceeded, an alarm is triggered.

If 15 minute average exceeds the STEL, a STEL alarm is generated.

The TWA alarm is generated when the current average of concentration, since the TWA was last cleared, has exceeded the TWA exposure limit.

During calibration, all alarms are disabled. Once the calibration is complete the alarms are re-enabled.

2.5. STEL, TWA, MAX and PEAK Operation

The 2020's meter display can be configured to show one of 4 values: STEL, TWA, PEAK and MAX.

2.5.1. Short Term Exposure Limit (STEL) Mode

The Short Term Exposure Limit (STEL) mode displays the concentration as a 15 minute moving average. 2020 maintains 15 samples, each representing a one minute averaging interval.

Once every minute, the oldest of the 15 samples is replaced with a new one minute average. This moving average provides a 15 minute average of the last 15 minutes with a one minute update rate. Since the average is calculated using 15 one minute averages, the meter display will only update once every minute.

STEL is set to zero each time the instrument is turned on. Since STEL is a 15 minute moving average, there is no need to clear or reset the STEL.

STEL calculations are always being performed by 2020. You can display the results of the calculations by selecting STEL as the Display mode. See Section 2.8.2 for details of switching between display modes.

2.5.2. Time Weighted Average (TWA) Mode

The TWA accumulator sums concentrations every second until 8 hours of data have been combined. If this value exceeds the TWA alarm setting, a TWA alarm is generated. The TWA is not calculated using a moving average. Once 8 hours of data have been summed, the accumulation stops. In order to reset the TWA accumulator, press the "Clr" key.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While you are in TWA mode, the time on the status display will show the number of minutes and hours of data that TWA has accumulated. When this reaches 8:00 or 8 hours, 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by 2020. You can display the results of the calculations by selecting TWA as the Display mode. See Section 2.8.2 for details of switching between display modes.

2.5.3. MAX Mode

The MAX mode displays the maximum signal, with the date and time that it was recorded. 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is displayed on the meter display.

The right soft key is used to clear the meter when displaying MAX. The "Clr" key only affects the reading that the meter is displaying. For example, if you display the MAX reading, and you press "Clr", only the MAX value is cleared. The TWA is still accumulating in the background.

2.5.4. PEAK Mode

The PEAK mode displays the current detected concentration. The reading is updated once a second. In the background, the 2020 datalogger is sampling the concentration and measuring minimum, maximum, and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the datalogger until the datalogger is full. See Section 2.8.2 for details of switching between display modes.

2.6. Set Functions

Set functions are used to setup 2020. There are 3 functions which can be set on the 2020; Calibration, Pump and Clock. Figure 6 shows a map detailing the Set functions and the key presses required for accessing them.

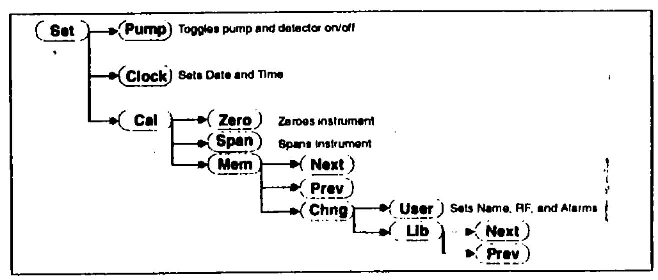

Figure 6 Set Function Map

2.6.1. Pump

The Pump function is used to control the pump. After selecting Set Pump, 2020 responds by displaying the new pump status.

The detector is also turned off when you turn the pump off. This prevents the detector from being damaged when there is no sample flowing through the detector.

When the pump and the detector are off, the meter display will be blank. Turn the pump and detector off when concentration measurements are not necessary, and 2020 will only be used for reviewing data or generating reports. By operating the instrument with the pump and detector off when you do not need them, you will conserve the battery and ultraviolet (UV) lamp.

1. Press the ENTER key. The top line of the status display changes to "Select?". The bottom line displays 3 soft key names; "Set", "Log" and "Disp".
2. Press the soft key below "Set".
3. The names of the soft keys change to reflect the Set options. The display now shows 3 devices which can be set: "Clock", "Pump", and "Cal". Press the "Pump" key.
4. The 2020 turns the pump off. If the pump was off, pressing "Pump" will turn the pump on.
5. A message will be displayed to show you the status of the pump. 2020 reverts back to the previous menu after a few seconds.
6. To return to the default display, press the ENTER key.

2.6.2. Clock

The Clock function is used to set both the current date and time.

1. Press the ENTER key.
2. Press the "Set" key.
3. When the names of the soft keys change, press the "Clock" key.

   The up and down arrows are used to change the character underlined by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is

   advanced past the right most character, it wraps around to the first character again.

   Formatting characters, such as the colon (:) in the time and the slash (/) in the date are skipped when advancing the cursor.

4. Use the "arrow keys" to enter the correct time. The time is formatted as Hour:Minute:Second.
5. Press the ENTER key to confirm the time and move to the date option.
6. When setting the date, the 2020 prompts you for the current date formatted as Year/Month/Day. Use the "arrow keys" to enter the correct date.
7. Press the ENTER key to confirm the date and return to the Set options. You can wait for the display to timeout or press ENTER to return to the default display.

2.6.3. Cal

Cal allows you to setup and calibrate 2020. You have three options under the Cal function: "Zero", "Span" and "Mem".

A calibration memory consists of a name, a response factor, and PEAK, TWA and STEL alarm levels.

The "Zero" and "Span" keys will be covered in detail in Section 3.1.

To edit the calibration memory , select "Mem" and then "Chng". The 2020 prompts you with two new soft keys; "User" and "Lib".

2.6.4. Library

Library selections simplify Cal Memory programming, and provide standard response factors for approximately 70 applications. "Lib" allows you to select an entry from a pre-programmed Library. The name, response factor and three alarm levels are all set from the library. To select a library entry to program the selected Cal Memory:

1. Select "Set", "Cal", "Mem", "Chng" and "Lib".
2. Use the "Next" and "Prev" keys to scroll through the list. See Appendix 8.7 for a list of the library entries.

3. When the required entry is displayed, press the ENTER key to select it. All the Cal Memory parameters will now be copied from the selected library entry to the current calibration memory.
4. Press the "Set", "Cal", "Mem", "Chng", "User" keys to review the Cal Memory settings.

   The "User" key is used to edit the Cal Memory information manually. Press ENTER to move you through each step of the cal memory: name, response factor, PEAK, STEL and TWA alarms. "User" will be covered in more detail in Chapter 3.

   You can change any of the values entered in the Cal Memory. Changes, made to the library information that has been loaded into a Cal Memory, will have no effect on the original library entry.
5. Press the ENTER key to proceed to the next step.

2.7. Log Functions

The Log functions provide options for reporting, editing and configuring 2020's built in datalogger. Figure 7 shows a map of the Log functions.

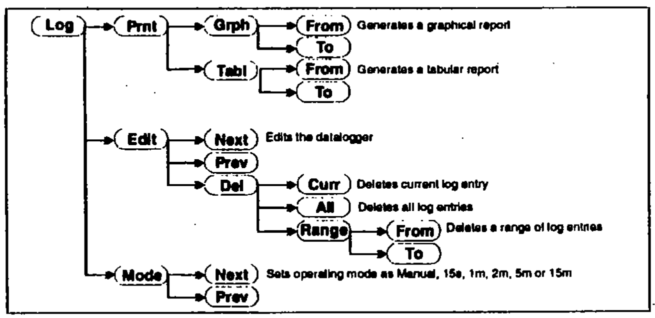

Figure 7 Log Function Map

2.7.1. Mode

2020 has two operating modes; manual and interval. When operating the 2020 in manual mode, each log entry contains a background and sample reading. In interval mode, 2020 automatically records the minimum, maximum and average readings

at the preset interval. The 2020 also records date, time and status with each entry in the datalogger.

You can set an averaging interval: 15 seconds, 1 minute, 2 minutes, 5 minutes or 15 minutes. 2020's datalogger can store 1000 entries. The interval you select will determine the period of operation. At the end of the period, the datalogger will be full and you will see an "Elog" status.

Averaging Interval	Hours of Operation to Fill the Datalogger
15 s	4.1
1 m	16
2 m	33
5 m	83
15 m	250

Table 2 Averaging Intervals and Periods

To change the datalogging mode:

1. Press the ENTER key.
2. Press "Log" and select the "Mode" key.
3. Use the "Next" and "Prev" keys to scroll through the list.
4. Press the ENTER key. If you select manual operation, the datalogger will be cleared.

When you switch between an averaging interval and manual operation, the datalogger will be cleared. Before the datalogger is cleared, 2020 will confirm your selection with the prompt "Are you sure?". Press "YES" to confirm your selection and clear the datalogger. If you do not want to lose your previously recorded data, press "NO", then print or save the data to disk before changing to manual operation. See Sections 2.7.3, and 4.2 for printing and saving logged data.

2.7.2. Edit

Edit allows you to review and delete the contents of the datalogger. To review the contents of the datalogger, use the "Next" and "Prev" keys. The average concentration and the time it was recorded are displayed. Hold down the keys to scroll through the data more quickly.

1. To delete entries from the datalogger, press "Del". You can delete the current entry, all entries or a range of entries.

Note:	Deleted information cannot be recovered. You should play back and print or download the contents of the datalogger before deleting any information.

2. To delete the current entry, press "Curr". You will be prompted to confirm your selection. Press "YES" to delete the last displayed entry. Press "NO" to return to the Delete options.
3. If you want to delete the entire contents of the datalogger, press "All". You will be prompted to confirm your selection. Press "YES" to delete all entries. Press "NO" to return to the Delete options.
4. If you want to delete a range of entries you must select a start and stop time. Press "Range" and use the "Next" and "Prev" keys to select the start time.
5. Press the ENTER key, and select the stop time. All entries between, and including, the start and stop entries will be deleted.

Note:	Deleting entries from the middle of the datalogger will not free space to store more entries. Deleting entries from the start of the datalogger, or at the end of the datalogger will free usable space.

2.7.3. Print

You can print logged data in tabular or graphical format. You must have a printer connected to 2020 in order to use the print options. See Section 4.1 for instructions on connecting and configuring a printer for use with 2020. See Section 4.3 for instructions on connecting the serial to parallel converter to 2020 and to the printer.

When you are using interval operation, the date, time and instrument status are printed along with the minimum, average and maximum concentration of each averaging interval. See Figure 8.


```
                 2020 Report

Date      Time     Status     Min     Avg     Max
95/03/07  11:24:21 OK         3.5     15.4    62.7
95/03/07  11:24:36 OK         2.7     8.8     46.3
95/03/07  11:24:51 OK         12.2    57.9    98.2
95/03/07  11:25:06 OK         2.9     53.1    98.5
95/03/07  11:25:21 OK         3.5     232     238
95/03/07  11:25:36 OK         40.7    360     496
95/03/07  11:25:51 ALPK       60.3    606     916
95/03/07  11:26:06 ALPK       129     943     1832
95/03/07  11:26:21 ALPK       4.9     705     1702
95/03/07  11:26:36 OK         3.4     3.9     5.7
```

Figure 8 Tabular Report (Averaging Interval)

The graphical report prints a range of entries from the datalogger. 2020 stores one set of readings (Min, Avg and Max) for each averaging interval. In each averaging interval the graphed minimum is the minimum of all the stored readings in that interval. The graphed Avg is the average of all the stored readings for the interval and the Max is the maximum of all the recorded maximum readings. In manual operation, the background, sample and the difference are printed.

A minus sign is used to draw the graph from the minimum to the average. Plus signs are used to graph the area between average and maximum. See Figure 9.

```
                          2020 Report

Date      Time     Status |0                                       2000|

95/03/07  11:24:21 OK      ++
95/03/07  11:24:36 OK      +
95/03/07  11:24:51 OK      -+
95/03/07  11:25:06 OK      -+
95/03/07  11:25:21 OK      ----+
95/03/07  11:25:36 OK      -------+++
95/03/07  11:25:51 ALPK     ----------++++++++
95/03/07  11:26:06 ALPK         ------------------++++++++++++++++++
95/03/07  11:26:21 ALPK    --      -------------+++++++++++++++++++++
95/03/07  11:26:36 OK      +
```

Figure 9 Graphical Report (Averaging Interval)

In manual operation the date and time are printed. A minus sign is used to draw the graph from the background to the difference. Plus signs are used to graph the area between difference and sample. Difference is calculated by subtracting the background from the sample.

1. Press the ENTER key. Select "Log", "Prnt" and then select "Grph" or "Tabl".
2. Select a start time using the "Next" and "Prev" keys. Hold down the "Next" and "Prev" to scroll through the data quickly.
3. Press the ENTER key, and select the stop time. All entries between, and including, the start and stop entries will be printed.

While printing, the 2020 displays a busy message. Do not try to perform additional functions while 2020 is printing. Pressing EXIT will abort the printout.

Although interval and manual mode generate different reports, the format of the reports is similar. For interval data, the column headings for the results are Min, Avg and Max. For manual data, the column headings are BkGd, Sample and Diff.

2.8. Display Functions

The Display function is used to control and configure 2020's display options. These options control the backlighting, select the display format, and lock the display to prevent access. Figure 10 shows a map of the Display functions.

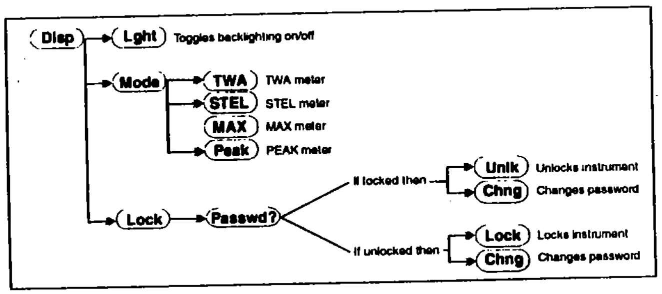

Figure 10 Display Function Map

2.8.1. Light

The Light function is used to switch the backlighting on and off.

1. Press the ENTER key.
2. Select "Disp" and then press "Lght". If the backlighting was off, it will be turned on.
3. Press EXIT to return to the function display.

To extend the operating life of the battery pack, turn the backlighting off when it is not required.

2.8.2. Mode

The Mode function is used to select the display type. You can select TWA, STEL, MAX, and PEAK.

1. Press the ENTER key.
2. Select "Disp" and then press "Mode". Select the mode you want to view. The current mode is not assigned a soft key.

   For example, if the current mode is TWA, then when you press the "Mode" key, "PEAK", "STEL" and "MAX" will be displayed.

When you have selected the new display mode, the meter display and status display will be updated to reflect the new mode. See Section 2.5. for a detailed description of each mode.

2.8.3. Lock

The "Lock" key is used to prevent access to 2020's sensitive options. Sensitive options are those which can affect 2020's readings. These include selecting a calibration memory, zeroing the instrument and spanning the instrument. If any one of these functions are selected when the instrument is locked, 2020 will prompt you to unlock the instrument before you can access the functions.

To lock 2020:

1. Press the ENTER key. Press "Disp" and then select "Lock".

2. You will be prompted for a password. If no password has been entered, then press the ENTER key. If a password has been entered, enter the correct password and then press the ENTER key.
3. If you want to enter a password, press "Chng" and enter a password. The password can be up to 4 numerical characters. Press the ENTER key to accept the password.

Note:	If you change the password, make sure you record the setting. Once the instrument is locked, there is no way to unlock it without losing all your data.

4. Press "Lock" to lock 2020. The message "Lock is on" will be displayed. If "Unlk" is displayed, 2020 is already locked.

2.8.4. Unlock

If you try to calibrate 2020 while it is locked you will see the message "Error: LOCKED! See Disp". To gain access:

1. Press EXIT twice and then press "Disp".
2. Press "Lock", enter the password and press ENTER. Once the password has been entered and verified, press "Unlk" to unlock the instrument.
3. Press EXIT twice continue with calibration.

3. Detailed Operation

3.1. General Information

2020 must be calibrated in order to display concentration in ppm or mg/m^3 units equivalent to the calibration gas. First, a supply of zero air which contains no ionizable gases or vapors, is used to set 2020's zero point. Then, calibration gas, containing a known concentration of a photoionizable gas or vapor, is used to set the sensitivity.

Occasionally clean ambient air will be suitable as zero air. Due to 2020's sensitivity, outdoor air is usually unsuitable for calibration. For best results, use a commercial source of zero grade air and a second regulator. Zero air should have not more than 0.1 ppm total hydrocarbons (THC).

If compound threshold limit values (TLVs) are exceeded, you should use a gas bag for sampling and calibration.

To determine the TLV of the compounds contained in the calibration gas, refer to the Material Safety Data Sheet (MSDS) supplied with your calibration gas cylinder.

If you will be using a gas bag for calibration, you should obtain the calibration kit (Photovac Part No. 390033). The calibration kit contains a regulator, a gas sampling bag and a gas bag adapter. See 3.3 for details of calibration using a gas bag.

Note: Disconnect 2020 from the AC adapter before beginning calibration.

3.2. Calibration Using the Flow-Match Regulator

3.2.1. Connecting the Flow-Match Regulator to the Cylinder

Warning: Observe proper handling procedure for all gases! See Section 1.2.2.

1. Connect the regulator to the calibration gas cylinder.

   If you are using a portable tank of calibration gas (Photovac Part No. 350012), connect the regulator (Photovac Part No. 350006) directly to the tank.

2. When the regulator is connected properly, you can read the cylinder contents from the regulator gauge.
3. Connect the adapter tubing to the regulator.

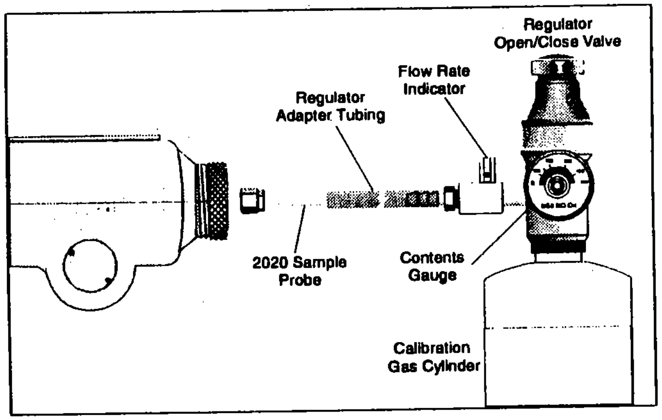

Figure 11 Flow-Match Regulator

3.2.2. Calibrating 2020 with the Flow-Match Regulator

1. Ensure the short sample probe is connected to the 2020 inlet. If you are using the long probe for sampling, then ensure the long probe is connected to 2020.

Note: Ensure the sample probe is free of any contamination as this will effect the calibration

2. Press the ENTER key.
3. Select "Set", "Cal" and then "Mem".
4. Select the desired Cal Memory. 2020 has 15 Cal Memories and can be calibrated with 15 different span gases or response factors if required. Only one Cal Memory can be used at a time. Each Cal Memory stores a different response factor, zero point, sensitivity, and alarm levels.
5. Select "Chng" and then "User". Enter a name for the calibration memory.

   Press the ENTER key and enter a response factor (RF). Refer to Appendix 8.6 for a list of Response Factors. If the compound is not listed in Appendix 8.6 or you are measuring gas mixtures, then enter a value of 1.0. The concentration detected by 2020 will be multiplied by the response factor before it is displayed and logged.

6. Press the ENTER key and enter an alarm level for STEL, TWA and PEAK.
7. Press ENTER and expose 2020 to a supply of zero air.
8. Select "Set", "Cal" and "Zero". Allow 2020 to set its zero point.
9. Select "Set", "Cal" and "Span". 2020 asks for the span gas concentration. Enter the known span gas concentration, without pressing the ENTER key to confirm it.
10. Insert the 2020 sample probe into the adapter tubing from the regulator. See Figure 11.
11. Ensure the calibration gas cylinder is upright and open the regulator by turning the valve counterclockwise. Open the regulator until the ball is 1/8" from its rest position.

Note: Do not set the flow rate too high.

12. Press the ENTER key. 2020 sets its sensitivity.
13. When the display reverts to the default display, 2020 is calibrated and ready for use.

14. Remove the adapter tubing from the inlet and close the regulator.

If you turn off 2020 in the middle of zeroing or spanning, the next time you turn it on it will display a Cal status. This indicates that you need to calibrate 2020.

Note:	While the Cal status is active, all alarms are deactivated.

3.3. Calibration Using a Gas Bag

3.3.1. Preparing the Calibration Gas Bag and the Zero Air Bag

Use the calibration kit (Photovac Part No. 390033) as follows:

Warning:	Observe proper handling techniques for all gases! See Section 1.2.2.

1. Connect the regulator to the calibration gas cylinder.

   If you are using a portable tank of calibration gas, connect the regulator (Photovac Part No. 600649) directly to the tank.

   If you are using a large cylinder of calibration gas, you must obtain a high purity regulator as specified in Section 1.4. Isobutylene in air is usually supplied with a standard CGA 590 cylinder valve outlet. Obtain a regulator with the matching fitting. Connect the regulator to the tank of calibration gas. Tighten the regulator onto the tank with a wrench. Do not over-tighten.

Note:	Do not force the connection. Do not use Teflon tape with CGA fittings. In general, these fittings are designed for metal to metal sealing. Do not use adapters to connect one CGA fitting to another type of CGA fitting. If the regulator does not match the outlet on your calibration tank, contact your specialty gas supplier.

2. Attach the knurled nut on the gas bag adapter to the regulator. Finger-tighten the fitting.

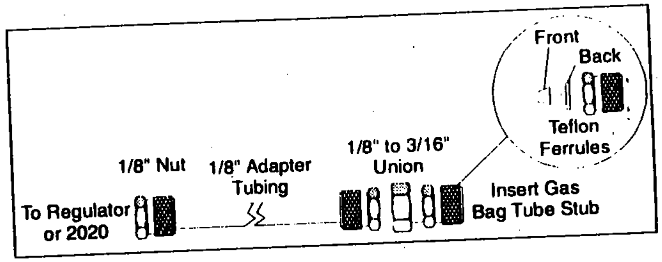

Figure 12 Gas Bag Adapter

3. Loosen the knurled nut on the reducing union of the gas bag adapter.

Note:	Do not remove the nut from the union as the Teflon ferrules contained inside the nut may be lost. See Figure 12.

4. Insert the tube stub from the gas bag into the knurled nut. Tighten the knurled nut and ensure the tube stub is secure. If the gas bag is not secure, ensure you have inserted the tube stub far enough into the knurled nut. Do not over-tighten the fitting.

Note:	Over-tightening the Teflon ferrules will result in damage to the ferrules!

5. The union should be connected to the gas bag adapter. If it is not, then tighten the nut on the adapter tube to the union.
6. Flush and fill the gas bag. See Appendix 8.5 for instructions.
7. Remove the knurled nut on the adapter tube from the regulator.
8. Repeat this procedure, if necessary, to prepare a bag of zero air.

Note:	Do not use the same gas bag or gas bag adapter for the bag of zero air. You will contaminate the bag of zero air.

3.3.2. Calibrating 2020 with a Gas Bag

1. Disconnect the probe from the 2020.
2. Press the ENTER key.

3. Select "Set", "Cal" and then "Mem".
4. Select the desired Cal Memory. 2020 has 15 Cal Memories and can be calibrated with 15 different span gases or response factors if required. Only one Cal Memory can be used at a time. Each Cal Memory stores a different response factor, zero point, sensitivity, and alarm levels.
5. Select "Chng" and then "User". Enter a name for the calibration memory.
6. Press the ENTER key and enter a response factor.
7. Press the ENTER key and enter an alarm level for each mode.
8. Press the ENTER key and enter a response factor. Refer to Appendix 8.6 for a list of Response Factors.

   If the compound is not listed in Appendix 8.6 or you are measuring gas mixtures, then enter a value of 1.0. The concentration detected by 2020 will be multiplied by the response factor before it is displayed and logged.
9. Connect the supply of zero air. If you are using a gas bag with zero air, open the bag and connect the gas bag adapter to the inlet.
10. Select "Set", "Cal" and "Zero". The 2020 sets its zero point.
11. Select "Set", "Cal" and "Span". The 2020 asks for the span gas concentration. Enter the known span gas concentration, without pressing the ENTER key to confirm it.
12. Open the bag and then connect the gas bag adapter to the inlet. Press ENTER. 2020 sets its response factor.

Note:	Readings may fluctuate slightly as the gas bag empties. Do not allow 2020 to evacuate the bag completely.

13. When the display reverts to the default display, 2020 is calibrated and ready for use. Remove the span gas bag from the inlet.

If the 2020 is powered off in the middle of zeroing or spanning, it will power on displaying a Cal status. This indicates that you need to calibrate 2020. While the Cal status is active, all alarms are inactive.

3.4. Programming the Cal Memories

2020 has 15 Cal Memories and can be calibrated with 15 different span gases or response factors if desired. To program the Cal Memories:

1. If you will be calibrating directly from the portable cylinder, connect a flow-match regulator (Photovac Part No. 350006) to each tank. You must use a separate regulator for each compound to prevent cross contamination.

   If you are using gas bags, prepare the bags of calibration gas as outlined in Section 3.3. Use a different gas bag and gas bag adapter for each concentration and for each type of calibration gas. You can use the same gas bag to zero all the Cal Memories, however, you must refill the bag for each Cal Memory.
2. Select "Set", "Cal" and "Mem".
3. Select the desired Cal Memory (1 to 15) with the "Next" and "Prev" keys.
4. Press "Chng" to change the parameters of the Cal Memory. Select "User" or "Lib".
5. If you selected "User", enter the name, response factor and alarm levels.
6. If you entered "Lib", use the "Next" and "Prev" keys to select the required library. See Appendix 8.7 for a list of Library entries.

Note:	It does not matter which Cal Memory is selected or which response factor is entered, 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

7. Calibrate the instrument as described in Section 3.2.2 or 3.3.2. When the calibration is completed, the calibration information is automatically stored in the selected Cal Memory.
8. Repeat this procedure for each Cal Memory you need.

Whenever the instrument is calibrated, 2020 updates the selected Cal Memory only. Each Cal Memory must be calibrated at least once a day. Frequency of calibration will depend on ambient conditions and instrument response. If ambient conditions change or the response has drifted, a calibration must be performed for each Cal Memory to ensure reliable operation.

3.5. Response Factors for Gases and Vapors

3.5.1. General Information

In situations where only a single pure compound is present in air, 2020 should be calibrated with a standard of that specific compound as span gas. 2020's 15 Cal Memories can be used to store calibration information for 15 different span gases.

The displayed reading will always be influenced by any other photoionizable compounds present in the air sample.

Note: Even if 2020 has been calibrated with a specific compound, its response is not specific and the presence of another ionizable impurity may render the numerical result invalid.

It is often impractical to carry a range of different standards into the field. Approximate results can be obtained by calibrating 2020 with the recommended span gas and entering the appropriate response factor. The response factor is based on the ratio of the response of the specific compound to the response of the span gas. The response factor multiplies 2020's reading then displays and records it.

Appendix 8.6 provides response factors from which approximations can be made for guidance purposes. Data extrapolated from the use of response factors must be regarded as interim and approximate only. Appendix 8.6 should be used only for concentrations up to 500 ppm of the specific compound, as response factors change with concentration.

3.5.2. Using Response Factors

1. Select "Set", "Cal" and "Mem".
2. Select the desired Cal Memory (1 to 15) with the "Next" and "Prev" keys.
3. Press "Set", "Cal" and "Mem" and "Chng" to change the parameters of the Cal Memory. Select "User" or "Lib".

4. If you selected "User", enter the name, response factor and alarm levels.
5. If you entered "Lib", use the "Next" and "Prev" keys to select the required library. See Appendix 8.7 for a list of Library entries.
6. Calibrate 2020 with zero air and 100 ppm isobutylene as described in Section 3.2.2 or 3.3.2.
7. Expose 2020 to the sample. The displayed reading is the approximate concentration of the specific compound.

The response factors in Appendix 8.6 serve only as a guide to concentrations measured by 2020.

Results are expected to be accurate to within +/-10 ppm or +/-25% of the displayed result, whichever is greater. Accuracy of response factors to other gases and vapors may differ from those listed in Appendix 8.6.

3.6. Manual Operation

As part of manual operation, you setup 2020 to monitor various locations. Since each location may contain different compounds and concentration ranges you can program a Cal Memory and the associated response factor and alarm level for up to 15 different applications. In this way you can sample numerous locations without having to re-calibrate 2020 at each location.

Prepare a monitoring schedule for you application. Your schedule should contain a list of sites that must be monitored and the Cal Memory that must be used when monitoring the site. Also include any reference information that will help you define the site and the monitoring application. If you create your schedule using spreadsheet software, you can later download 2020 data to a computer and then copy it into the spreadsheet for further calculations.

Once you have programmed 2020, and prepared a list of sites to be monitored, you will move around to each location and manually log data at each site.

1. You must determine the number of calibration standards that will be required to perform manual monitoring for your application. Program and calibrate all the calibration memories that you need. See Section 3.2 and 3.4.

Note:	It does not matter which Cal Memory is selected or which response factor is entered, 2020's response is not specific to any one compound. The reading displayed always represents the total concentration of all ionizable compounds in the sample.

2. Ensure the 2020 is in PEAK mode. To change the mode, press ENTER and select "Disp". Press "Mode" and select PEAK.
3. Switch to manual operation. Press ENTER and then "Log". Select "Mode". Use the "Next" and "Prev" keys to scroll through the list. When Manual is displayed press ENTER. When you switch between interval and manual operation, the datalogger will be cleared. Press "YES" to confirm your selection and clear the datalogger. If you do not want to lose your previously recorded data, press "NO", then print or save the data to disk before changing to manual operation. See Sections 2.7.3 and 4.2 for printing and saving logged data.
4. The instrument status will change to "Loc".
5. Select the required Cal Memory for this location. Press ENTER and select "Set", "Cal" and then "Mem". Use the "Next" and "Prev" keys to select the desired Cal Memory.
6. Press the ENTER key and locate the first site on your schedule. The middle soft key is used to advance to the next measurement when you are operating in manual mode. Press the "Next" key. If you are not using manual operation, the "Next" key is not shown.
7. The instrument status will change to "BkGd". A background measurement must be made. When you have an accurate background, press "Next". 2020 will record the displayed concentration when you press the "Next" key.
8. The instrument status will now be "Samp". Take a sample measurement. When you have an accurate sample, press "Next". 2020 will record the displayed concentration when you press the "Next" key.
9. The instrument status will again be "Loc". Go to the next site on your schedule.

When you have completed your monitoring you can download the contents of the datalogger to a computer and then add the 2020 data to your spreadsheet.

If you change from manual operation to an averaging interval, you will lose the contents the datalogger. Print or save the data to disk before changing the interval. See Sections 2.7.3 and 4.2 for printing and saving logged data.

3.7. Preparing for Field Operation

3.7.1. Field Check List

When using 2020 for field operation, the following items should be carried into the field to reduce or eliminate down time of the instrument.

If you are going to be in the field for a single 8-10 hour day, then you should include the following accessories:

- Spare battery pack (Photovac Part No. 350009)
- Spare UV lamp ((Photovac Part No. 390011)
- 2020 multi-tool (Photovac Part No. 396012)
- Sample line (Photovac Part No. 390006)
- Calibration kit(s) (Photovac Part No. 390033)
- Calibration regulator (Photovac Part No. 350006)
- Tank(s) of calibration gas (Photovac Part No. 350012)
- Spare gas bag for zero air (Photovac Part No. 396017)
- Gas bag adapter for zero air (Photovac Part No. 396010)
- Supply of commercial zero air
- Spare inlet filters (Photovac Part No. 396015 or 396000)
- Dilution probe (Photovac Part No. 350013)
- Spare charcoal filters for the dilution probe (Photovac Part No. 395064 or 395067)
- Carrying case (Photovac Part No. 350010)
- User's manual (Photovac Part No. 350001)
- DC power cord (Photovac Part No. 350004)

Table 3 Check List for Field Operation

If you will be in the field for more than one day you should include the following additional items:

- AC adapter (Photovac Part No. 350001 or 396013)
- Printer (Photovac Part No. 380120)
- Cable kit (Photovac Part No. 350011)
- Computer and associated cables
- Serial to parallel converter (Photovac Part No. 380145)

Table 4 Additional Field Items

3.7.2. Operational Check List

Before beginning field work, set up and calibrate 2020 for your particular application. Ensure the instrument is in working order before heading into the field:

1. Press the "Set" and "Clock" keys and ensure the correct time is entered. Press the ENTER key and ensure the correct date has been entered.
2. Ensure the battery pack is fully charged. If you are unsure about the status of the battery, replace the battery pack with one that is fully charged. See Section 1.5.
3. Program and calibrate all the Cal Memories you need. See Section 3.4. After calibration is complete, sample the calibration gas and the bag of zero air to ensure 2020 has been calibrated correctly.
4. Select the correct operating mode. See Section 2.8.2.
5. Reset the TWA accumulator, the STEL moving average and the MAX. See Section 2.5.
6. You may want to delete all entries from the datalogger to avoid confusion between different days' data and to avoid running out of space in the datalogger. See Section 2.7.2.

4. Connecting Accessories

4.1. Printer

The printer must have a printing width of at least 65 characters and must use fixed spaced fonts. The serial communication parameters on 2020 have been fixed at 9600 baud, no parity, 8 data bits and 1 stop bit. Your printer must be able to match these settings.

If you are using a parallel printer, you will need the Photovac serial to parallel converter (Photovac Part No. 380145). See Section 4.3 for details of connecting and operating 2020 with a serial to parallel converter.

Note:	2020 I/S is not classified for use in hazardous locations with a printer.

1. Turn 2020 off.

Note:	You must turn the instrument off before connecting or disconnecting the printer cable.

2. Connect the printer cable (Photovac Part No. 350011) to the I/O port on the back of the 2020 and then to the serial port on the printer.
3. Turn 2020 on.

4. The serial communication parameters on 2020 have been fixed. It communicates at 9600 baud, no parity, 8 data bits and 1 stop bit. You must set your printer accordingly.
5. Use the "Grph" and "Tabl" keys as detailed in Sections 2.7.3.

If this arrangement does not produce the desired results, see Section 6.4.

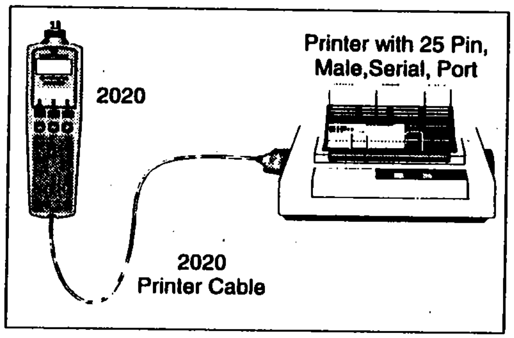

Figure 13 Connecting 2020 to a Serial Printer

4.2. Computer

2020 can send information stored in its datalogger to a computer. This option may be used if you need to prepare reports based on 2020's recorded data. This feature may also be used if you need the recorded data in a format that can be imported into a spreadsheet or database for further calculations.

Note:	2020 I/S is not classified for use in hazardous locations with computers.

You may need to keep more data than can be logged by 2020. If this is the case you have two options: you may print the data before they are deleted from the datalogger and keep the printed reports, or you may store the data on disk for later use. You may not, however, load data that have been stored on disk back into 2020.

In order to establish communication with 2020 and receive data stored in the datalogger, the computer must be set up to emulate a terminal. In order to do this you will need a communications software package that will allow you to transfer data from 2020 to a

computer. Software packages such as Crosstalk, and Procomm are recommended for use with 2020. Crosstalk XVI will be used as an example in the instructions below.

If you are using Microsoft Windows you do not need to purchase any separate software. Instructions for downloading information to Windows' Terminal program are also provided.

If you are already using another type of communication or terminal emulation software package, it is not necessary to purchase a copy of Crosstalk or Procomm. Use your communications package to set up the computer to receive data at 9600 baud, no parity, 8 data bits and 1 stop bit. Refer to the user's manual, provided with your software, for specific details.

Once data have been transferred to Crosstalk, they must be saved to a floppy or hard disk. In order to manipulate the data, you must import the data either as a text file into a spreadsheet, where the data may be parsed and converted to numeric data, or into a text editor or word processor where the data may be viewed and edited.

The instructions below will provide you with the most basic information for using 2020 with communications software. Please refer to the software user's manual for specific details of operation.

4.2.1. Using Crosstalk

The following instructions are for Crosstalk XVI Version 3.71. The commands may vary with the version of Crosstalk you are using. To initiate communications between 2020 and the computer:

1. Start Crosstalk. The Status Screen will appear.
2. At the bottom of the Status Screen there will be a highlighted bar with the word Command?. If the word Command? does not appear press the <Esc> key on the computer keyboard.
3. Type *SP* and press <Enter>. This will allow you to set the baud rate at which the computer will receive data from 2020. Type 9600 and press <Enter>.
4. On 2020 the number of data bits has been fixed at 8, stop bits has been fixed at 1. You must set up Crosstalk accordingly. Type *DA* and press <Enter>. Type in *8* and press <Enter>.
5. Type in *ST* and press <Enter>. Type in *1* and press <Enter>.

6. Type in *PA* and press <Enter>. Type in *None* and press <Enter>. This sets the parity to None
7. Turn 2020 off.

Note:	You must turn the instrument off before connecting or disconnecting the printer cable.

8. 2020 must be connected to a serial port. Use the printer cable (Photovac Part No. 350011) to connect 2020 to one of the computer's serial ports. Take note of which serial port you are using. Normally you will use Com1 or Com2.
9. If the 2020 printer cable plugs directly into the port on the computer without the gender changer or the 9 to 25 pin adapter (null modem), you are most probably connected to a parallel port. You will need at least one of the adapter cables to connect 2020 to a serial port. See Section 6.6.
10. Type *PO* and press <Enter>. Type in *1* if you connected 2020 to Com1 or type in 2 if you connected 2020 to Com2 and press <Enter>. If you connected 2020 to another serial port enter the corresponding number.
11. Type *MO* and press <Enter>. Type in *A* to change the mode to answer.
12. In order to capture the data and store them on a disk you must turn the Crosstalk capture command on and specify a disk to which the data can be stored. Type *CA* and press <Enter>.
13. Now type in the disk drive and the name of the file where you want the data stored. For example if you want to store the data in a file called 2020 on a floppy disk in drive A, then type *A:\2020.dta* and press <Enter>.
14. If the word Command? does not appear at the bottom of the Crosstalk Capture Screen, press <Esc>.
15. You have now set up Crosstalk to communicate with 2020. Type *GO L* to begin the downloading session. The Status Screen will disappear and the Capture Screen will appear.
16. Press the ENTER key. Select "Log", "Prnt" and then select "Grph" or "Tabl".
17. Select a start time using the "Next" and "Prev" keys. Hold down the "Next" and "Prev" to scroll through the data quickly.

18. Press the ENTER key, and select the stop time. All entries between, and including, the start and stop entries will be sent to the computer.
19. When all the data have been sent, press <Esc>. Type *CA off* and press <Enter>. This will turn the capture option off and write the captured file to the disk drive you specified in step #13.
20. Press <Home> to return to the Status Screen.
21. Type *QU* and press <Enter>. This will end your communications session.

Convert the file to a text file or a print file using the MS-DOS® Copy Command:

22. To convert the file to a text file, at the DOS prompt type: *Copy 2020.dta 2020.txt*
23. To convert the file to a print file, at the DOS prompt type: *Copy 2020.dta 2020.prn*

Once the file has been converted to a text file you can use a text editor to view and edit the contents.

If you are not comfortable with the Crosstalk commands used above, you should familiarize yourself with these commands by reading the Crosstalk user's manual. It is important to understand each of the commands in order to ensure both Crosstalk and 2020 are set up correctly.

4.2.2. Using Microsoft Windows

If you are using Microsoft Windows you do not need to purchase any separate software. The rules for connecting 2020 to the serial port still apply. See steps #7 to 9 in Section 4.2.1.

In order to use these instructions, you must be familiar with Microsoft Windows Version 3.1 and it must be installed and running on your computer.

To initiate communications between 2020 and Windows:

1. Start Windows and then start the Terminal Program. The Terminal Program may be in the Accessories Program Group. Its location will depend on how you have set up Windows.

2. Open the Settings Window and select Communications.

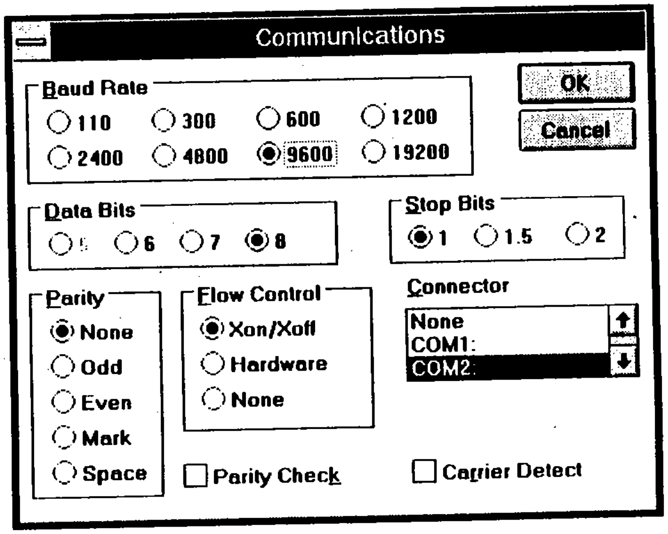

Figure 14 Terminal Program's Communication Dialog Box

3. Select the correct Baud Rate, Data Bits, Stop Bits, Parity and Connector. Select Xon/Xoff for the Flow Control option. Leave the Parity Check and Carrier Detect options off. Select OK to close the dialog box and accept the changes.

4. Open the Transfers Window and select Receive Text File. A Receive Text File dialog box will open. See Figure 15.

5. Type in the desired path and filename for the data that are to be downloaded. Ensure the path is correct.

   Ensure the file name has a .txt extension. The .txt extension will make it easier to use the downloaded data later.

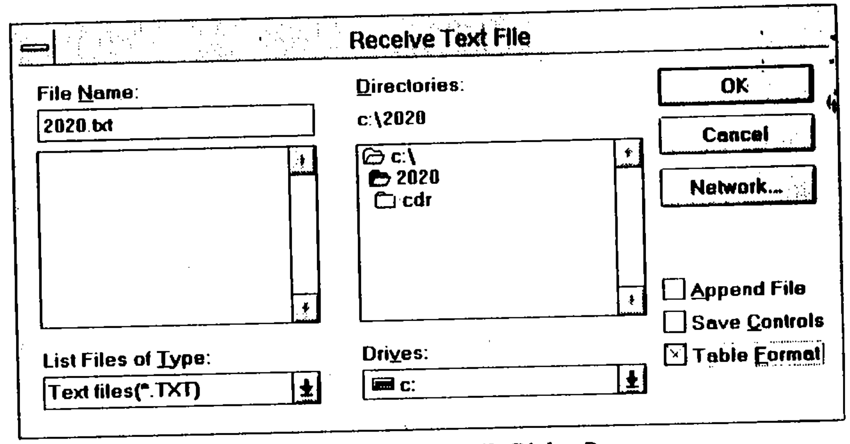

Figure 15 Terminal Program's Transfer Text File Dialog Box

6. Select the Table Format option and then select OK. At the bottom of the screen the message, "Receiving Data" is displayed.

7. Press the ENTER key. Select "Log", "Prnt" and then select "Grph" or "Tabl".

8. Select a start time using the "Next" and "Prev" keys. Hold down the "Next" and "Prev" keys to scroll through the data quickly.

9. Press the ENTER key, and select the stop time. All entries between, and including, the start and stop entries will be sent to the computer.

10. To terminate communications select Stop from the bottom of the screen.

The logged data can now be copied or cut to the Windows Clipboard and pasted into a text editor, such as Windows' Notepad, or a spreadsheet for editing.

Refer to the Microsoft Windows User's Guide for detailed instructions on installation and operation of Microsoft Windows.

4.3. Serial to Parallel Converter

This device allows 2020, which is only capable of serial communication, to communicate with a parallel device. This will be most useful for printing, as most printers utilize parallel communications.

The printer must have a printing width of at least 65 characters and must use fixed spaced fonts. The serial communication parameters on 2020 have been fixed at 9600 baud, no parity, 8 data bits and 1 stop bit. Your converter must be set to match these settings.

4.3.1. DIP Switch Settings

The DIP switch settings are very important and must be set correctly in order to correctly communicate with 2020. 2020 will always be the serial device so the data flow direction will be serial to parallel.

To configure the serial port of the converter:

1. Locate the DIP switches located on the side of the converter.

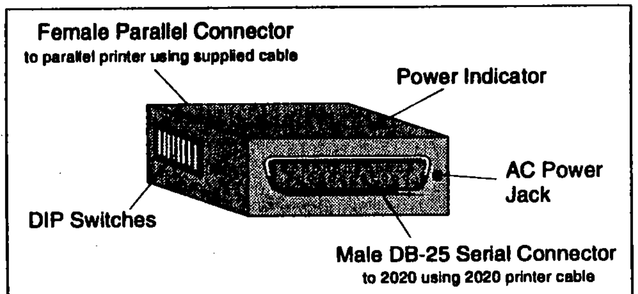

Figure 16 Layout of Serial to Parallel Converter

2. Determine the baud rate at which the converter will be receiving data from the instrument. The value you set here must match 2020.
3. Use the first three DIP switches to set the baud rate to 9600. See Table 5.

Baud Rate	SW1	SW2	SW3
9600	On	Off	On

Table 5 DIP Switch Settings for Baud Rate

4. Switch 4 sets the data bits. Off = 7 Bits. On = 8 Bits. Set the data bits to 8.
5. Use Switches 5 and 6 to set the parity to none.

Parity	SW5	SW6
None	Off	Off

Table 6 DIP Switch Settings for Parity

6. Switch 7 sets the Handshake mode. Off = Xon/Xoff. On = DTR. Set the Handshake mode to Xon/Xoff.
7. Use switch 8 to set the data flow direction to serial to parallel. Off = Serial to Parallel. On = Parallel to Serial.
8. Check your settings. It is imperative that you have the correct settings or you will be unable to establish communications. Your settings should match those in Table 7.

SW1	SW2	SW3	SW4	SW5	SW6	SW7	SW8
On	Off	On	On	Off	Off	Off	Off

Table 7 DIP Switch Settings

4.3.2. Using 2020 with the Serial to Parallel Converter

Note:	2020 I/S is not classified for use in hazardous locations with printers or with the serial to parallel converter. Turn 2020 off.

Note:	You must turn the instrument off before connecting or disconnecting the printer cable.

1. Unplug the converter from the AC outlet.
2. Use the cable kit (Photovac Part No. 350011) to connect the 2020 I/O connector to the converter.

3. Use the parallel cable, supplied with the converter, to connect the converter and the printer. If this cable is not suitable, see Section 6.5 for the cable requirements.

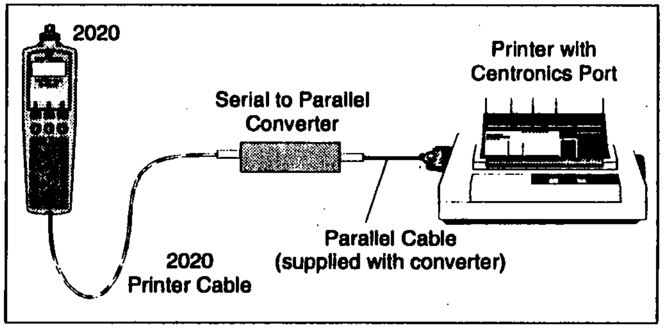

Figure 17 Connecting 2020 to the Serial to Parallel Converter

4. Plug the converter into an AC outlet.
5. Turn 2020 on.
6. Ensure the printer has been setup correctly. It must be on-line. Ensure paper is available and has been aligned properly.
7. Press the ENTER key. Select "Log", "Prnt" and then select "Grph" or "Tabl".
8. Select a start time using the "Next" and "Prev" keys. Hold down the "Next" and "Prev" keys to scroll through the data quickly.
9. Press the ENTER key, and select the stop time. All entries between, and including, the start and stop entries will be sent to the printer.

Sample Line

A 3 meter (9') sample line (Photovac Part No. 390006) may be connected to 2020 for remote sampling. Connect the sample line to the 2020 inlet using the fittings supplied with the sample line.

Note:	When using the sample line, be especially careful not to aspirate liquids or solids as they will damage 2020.

4.5. Dilution Probe

4.5.1. Description

The dilution probe (Photovac Part No. 350013) attaches to the underside of 2020 and allows 2020 to read concentrations up to 20000 ppm isobutylene equivalent units.

A metering valve controls the ratio of sample to zero air. 2020's pump draws air through both the dilution probe inlet and through the charcoal filter. You set the metering valve so that 2020 reads 10% of the actual sample concentration. Zero air is created by drawing room air through a charcoal filter.

2020 is calibrated with 100 ppm isobutylene. The dilution probe is then connected to the 2020 inlet and the dilution probe—2020 system is calibrated with 100 ppm isobutylene. For high accuracy operation, the dilution probe is calibrated with 1000 ppm isobutylene.

4.5.2. Assembling the Dilution Probe

1. Slide the charcoal filter tube into the 1/4" compression nut on the zero air inlet of the dilution probe. See Figure 18.

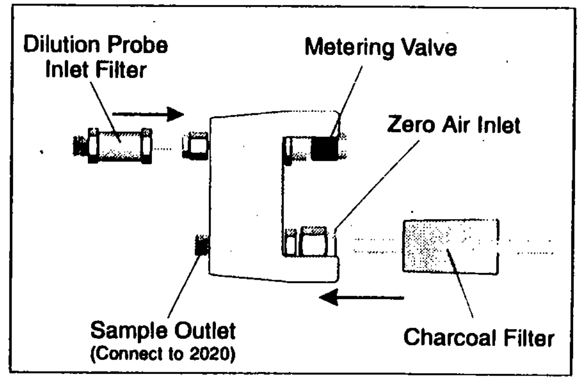

Figure 18 Dilution Probe Layout

2. Finger-tighten the nut. Then use a wrench to tighten the nut 3/4 of a turn further. This will compress the ferrules onto the tube.

3. Slide the inlet filter into the 1/8" compression nut on the sample inlet of the dilution probe. See Figure 18.
4. Finger-tighten the nut and then use a wrench to tighten the nut 3/4 of a turn further. This will compress the ferrules onto the filter tube.

4.5.3. Installing the Dilution Probe

1. Remove the two screws from the bottom of the 2020 control housing.
2. Use the long mounting screws supplied with the dilution probe to connect the mounting bracket to the 2020 bottom housing. See Figure 19.
3. Use the short mounting screws and the washers to connect the dilution probe to the mounting bracket.

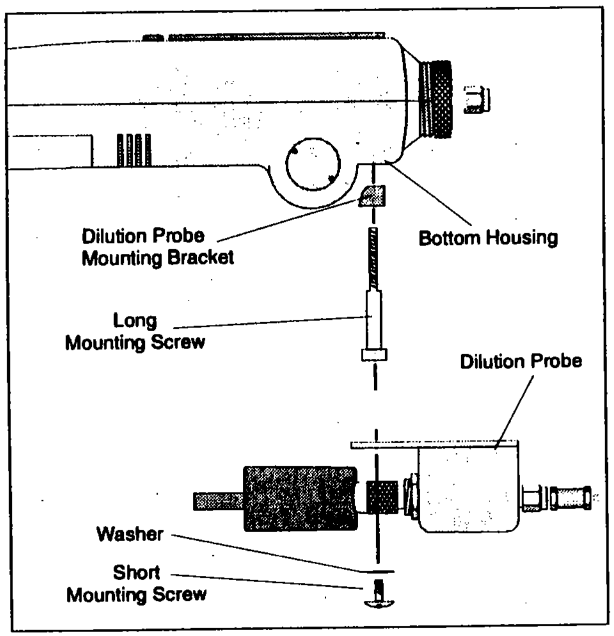

Figure 19 Installing the Dilution Probe

4.5.4. Calibration with the Dilution Probe

2020 must be calibrated in order to display concentration in units equivalent to ppm.

Occasionally clean ambient air will be suitable as zero air. If there is any doubt, use a commercial source of zero grade air and a regulator or second sampling bag. Span gas of the desired compound and concentration is also required and may be obtained from a specialty gas supplier. See Appendix 8.4.

Isobutylene at 100 ppm in air is recommended as span gas (Photovac Part No. 350012). If you are using another type of span gas, you must obtain the required gas and concentration. If compound threshold limit values (TLVs) are exceeded, you should use a gas bag for calibration.

Calibrate the instrument as follows:

1. Calibrate 2020 as outlined in Section 3.2.2 or 3.3.2.
2. Remove the 2020 sample probe. Use the tubing supplied with the dilution probe to connect the dilution probe outlet to the 2020 inlet.
3. If you are using gas bags, flush and fill the gas bags with 100 ppm isobutylene.
4. Use the hex wrench to loosen the screw in the metering valve handle. Do not remove the hex screw.
5. Insert the tube from the dilution probe inlet into the adapter tubing from the flow-match regulator. If you are using gas bags, connect the gas bag adapter to the dilution probe inlet.
6. Ensure the calibration gas cylinder is upright and open the regulator until the float is 1/8" from its rest position. Do not set the flow rate too high.
7. Adjust the metering valve until the display reads 10% of the actual span gas concentration.
8. Use the hex wrench to tighten the screw and lock the metering valve handle.
9. The dilution probe is now calibrated and ready for use. Disconnect the adapter tubing or the gas bag from the inlet.

Note:	When the dilution probe is connected to 2020, the displayed readings are always 10% of the actual reading.

4.5.5. High Accuracy Operation

The 2020–dilution probe system can be calibrated for higher accuracy operation. 2020 is calibrated with 100 ppm isobutylene and the dilution probe is calibrated with 1000 ppm isobutylene.

1. Calibrate the 2020 as described in Section 3.2.2 or 3.3.2. Use 100 ppm isobutylene as the calibration gas.
2. Connect the dilution probe to the 2020 inlet.
3. If you are calibrating directly from the calibration tank, Insert the 2020 sample probe into the adapter tubing of the regulator.

   Ensure the calibration gas cylinder is upright, and open the regulator by turning the valve counterclockwise. Open the regulator until the ball is 1/8" from its rest position. Do not set the flow rate too high.
4. If you are using gas bags, flush and fill the gas bags with 1000 ppm isobutylene. It is advisable to obtain a second calibration kit (Photovac Part No. 390033) for the 1000 ppm isobutylene. See Section 3.3.1 for details of filling the gas bags.
5. Adjust the metering valve until the display reads 10% of the actual isobutylene gas concentration.

4.6. Replacement Detector Lamps

4.6.1. General Information

2020 is supplied with a UV lamp which produces an energy of 10.6 electron-volts (eV). With this standard lamp installed, 2020 responds well to gases and vapors which ionize at 10.6 eV or less. Compounds that have an IP greater than 10.6 eV may not respond well to a 10.6 eV lamp.

For special applications, 2020's response can be changed by using lamps of other energies. The 11.7 eV lamp is covered in Section 4.6.2. You can obtain more information on lamps of other energies from the Photovac Applications Group.

4.6.2. 11.7 eV UV Lamp

With an 11.7 eV lamp installed, 2020 functions as a leak detector responding to gases and vapors which ionize at 11.7 eV or less. The 11.7 eV lamp may be useful for detecting leaks of chemicals not ionized by the 10.6 eV lamp. This lamp is intended for special applications only. It is not suitable for normal operation, because of limitations of the lamp window material.

The 11.7 eV lamp window material is Lithium Fluoride (LiF). Unlike other lamp windows, LiF readily absorbs water from atmospheric humidity. When contaminated by moisture, the window loses its ability to transmit UV light.

Note:	Never touch the window or let liquid water near it.

LiF is composed of two light elements which are easily disrupted within the crystal lattice by the UV light generated by the lamp. Disruption of the lattice causes the crystal to turn a yellowish color, and again performance declines.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Because of the lamp window limitations, the lifetime of the 11.7 eV lamp is restricted and it must be used sparingly according to the following instructions:

1. Remove the 11.7 eV lamp from the supplied dessicant bottle and install the lamp according to the instructions in Section 5.2.1.
2. Calibrate 2020 as outlined in Section 3.2 or 3.3.
3. Recalibrate 2020 every 15 minutes of operation.
4. Every hour of operation, switch off 2020 and examine the lamp window for yellowing. If the window is yellow, then remove the lamp and regenerate the window according to the procedure in Section 4.6.3.
5. After use, remove the lamp from 2020 and store it in the supplied dessicant bottle.

4.6.3. Regenerating the LIF Window

To regenerate the 11.7 eV lamp window:

1. Clean the lamp window with dry aluminum oxide powder on a dry cotton swab. Do not use methanol or water.
2. Aluminum oxide may be obtained from most chemical supply companies. When ordering specify 3.0 micron powder.
3. The lamp window can also be regenerated by storing the lamp in a dessicator for at least 5 days.

4.7. DC Power Cord

2020 can be connected to a car battery through the cigarette lighter with the DC power cord. While 2020 is connected to the car battery the 2020 battery is being charged.

Note:	2020 I/S is not classified for use in hazardous locations with a DC power cord.

1. Turn the instrument off by pressing the ON/OFF key for 2 seconds.
2. Connect the DC power cord (Photovac Part No. 390004) to the 2020 AC adapter jack on the rear of the instrument.
3. Connect the other end of the DC power cord to the cigarette lighter in the car.
4. Turn the instrument on again by pressing the ON/OFF key.

If the car is running, ensure the car exhaust does not contaminate your samples.

4.8. Belt Clip Holster

Use the belt clip holster (Photovac Part No. 350008) to protect the instrument and to mount the instrument to a belt or personal apparatus.

1. Disconnect the top strap and insert 2020 into the holster.
2. Reconnect the strap so that 2020 is held securely.

5. Routine Maintenance

5.1. Battery Charging

A fully charged battery pack powers 2020 for approximately 8 hours. If the instrument is to be used for more than 8 hours, carry a spare battery pack (Photovac Part No. 350009). When the first one has been discharged, replace it with the spare.

Note:	If you do not turn 2020 off before removing the battery pack, you will reset the instrument and you will lose all logged data and setup parameters.

When the instrument status displays "LBat", the battery pack requires charging. When the "LBat" status is displayed, you have 30 minutes of operation left. 2020 will turn itself off before the battery pack becomes critically low.

Note:	Do not remove or recharge the battery pack in a hazardous location.

Upon return from field work, charge the battery packs as outlined in Section 1.5. Use only the AC adapter specified for use with 2020.

If you do not require portable operation, you can use 2020 while it is connected to the AC adapter

The AC adapter automatically charges at a high charge rate until the battery pack is fully charged. It then maintains the full charge with a low continuous charge rate indefinitely so there is no danger of over-charging.

When the LED, on the back of 2020, is red, the battery is charging. When the LED turns green, the battery is fully charged and ready for use.

Note:	Leaving 2020 for more than 3 months without turning it on may result in the loss of recorded data and setup parameters. If 2020 is not used for long periods of time, turn the instrument on for a few hours every month to avoid loss of data.

2. Maintenance of the UV Lamp

5.2.1. Removing and Replacing the UV Lamp

Note:	Do not remove or replace the detector lamp in a hazardous location.

1. Ensure the instrument is turned off.

Warning:	You must turn the instrument off before removing the lamp cover!

2. Use the 2020 multi-tool to remove the lamp housing cover.
3. Tilt 2020 slightly and remove the UV lamp.

Warning:	Do not touch the wire grid inside the detector cell. Any dust or dirt in the detector cell can be blown out with a gentle jet of compressed air. Do not insert any object, other than the UV lamp, into the lampholder.

4. Discard the o-ring and spring supplied with the replacement lamp.
5. Without touching the lamp window, place the new lamp into the 2020 lampholder, window first. See Figure 20.

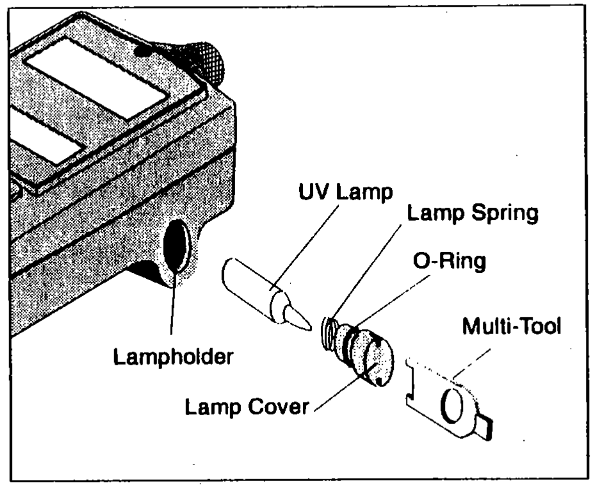

Figure 20 Removing the UV Lamp

Note:	If you have a UV lamp with a white serial number label, it is possible that the UV lamp may not fit into the lampholder. Do not force the lamp into the lampholder. See Section 6.3.

6. Replace the lamp housing cover. Tighten the cover down with the multi-tool. Do not overtighten.
7. Calibrate all the Cal Memories that you are using and then continue normal operation.

5.2.2. Cleaning the UV Lamp Window

During the course of normal operation, a film builds up on the window of the UV lamp. The rate at which the film develops depends on the type and concentration of the gases and vapors being sampled and results from the UV light interacting with them.

Hot gases and vapors may contribute to a decrease in sensitivity because they may condense on the lamp window. Condensation may eventually evaporate off the window, but it will usually leave a residue that must be removed by cleaning the lamp window.

As a guide, clean the window every 24 hours of operation.

Note: Do not remove the detector lamp in a hazardous location.

1. To remove the film, gently rub the window of the lamp with a lint free tissue moistened with methanol. Use only HPLC grade or spectroscopic grade methanol to clean the lamp window.
2. Allow the window to dry and then, without touching the window, place it back into 2020.
3. Calibrate all the Cal Memories that you are using and then continue normal operation.

5.3. Replacing the Sample Inlet Filter

2020 is equipped with a combined dust and water filter to reduce detector contamination. As the filter collects dust, 2020's inlet flow rate and sensitivity decrease. The filter will not allow water to pass through, but the filter will not stop all solvents.

Note: Do not aspirate liquid samples with 2020!

Replace the filter on a weekly basis, or more frequently if 2020 is used in a dusty or wet environment. You must replace the filter if 2020 has been exposed to liquid water. If you are sampling hot gases or vapors, condensation in the sample line may also affect the filter. The pump will sound labored when the filter requires replacement.

Note: Do not replace the inlet filter in a hazardous location.

1. Turn the instrument off. Unscrew the filter housing from the detector housing. Be careful not to lose the O-ring seal.

Note: Each filter is protected by a piece of blue plastic. Remove the plastic before installing the filter in 2020.

2. Remove the Teflon/Polypropylene filter and install the new filter (Photovac Part No. 396000 or 396015). Place the filter so that the Teflon side is facing down in the filter housing and the mesh side is facing the 2020.

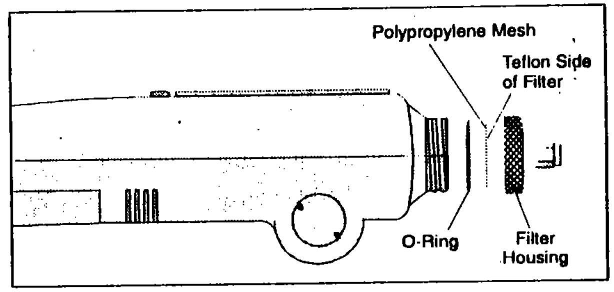

Figure 21 Replacing the Inlet Filter

Handle the filter disk only by the edges. The mesh may be damaged or contaminated by excessive handling. Use forceps if possible.

3. Replace the filter housing.
4. Calibrate all Cal Memories, that you are using, and then continue normal operation.

Warning: Do not operate 2020 without an inlet filter.

5.4. Maintenance of the Dilution Probe

5.4.1. Charcoal Filter

The charcoal filter will remove hydrocarbon contaminants for up to 4000 ppm-hours. This means that the filter will last for 1 hour removing 4000 ppm of hydrocarbon contaminants or will last for 4 hours removing 1000 ppm. The exact time will be determined by the operating environment. You will notice an increased hydrocarbon background when the filter requires replacement.

To replace the charcoal filter:

1. Loosen, but do not remove, the 1/4" compression fitting on the dilution probe zero air inlet and remove the charcoal filter from the dilution probe. See Figure 18 Dilution Probe Layout
2. Slide the replacement charcoal filter tube into the compression nut. (Photovac Part No. 395067).
3. Finger-tighten the nut. Then use a wrench to tighten the nut 3/4 of a turn further. This will compress the ferrules onto the tube.

When the dilution probe is not in use, place the charcoal filter in its plastic bag and store it in a clean, dry place.

5.4.2. Inlet Filter

The dilution probe is equipped with a dust filter to reduce detector contamination. As the filter collects dust, the inlet flow rate and sensitivity decrease. Replace the filter every 240 hours of operation, or more frequently if the instrument is used in a dusty environment.

Note:	Do not operate 2020 or the dilution probe without an inlet filter.

1. Turn 2020 off and remove the dilution probe from the instrument.
2. Hold the filter housing near the housing with a 9/16" wrench. See Figure 22.
3. Unscrew the top of the filter housing with another 9/16" wrench. Be careful not to lose the filter spring.
4. Remove the spring and filter. Install the new filter (Photovac Part No. 395000), open end first. Press the filter into place so it will seal at the bottom of the filter housing. Replace the filter spring and the top of the filter housing.

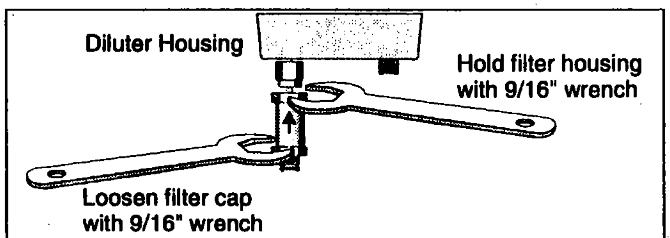

Figure 22 Removing the Dilution Probe Inlet Filter

5. Tighten the top nut while holding the bottom one stationary with the wrench.
6. Calibrate both 2020 and the dilution probe before continuing normal operation. See Section 4.5.4

6. Troubleshooting

6.1. General Information

If you have a service related question about 2020, consult this manual first. If you cannot find the answer in this documentation, contact the Photovac Service Department.

When you call, you should have your 2020 in front of you. You should also have this manual at hand. Lastly, please have the following information ready:

1. A description of what happened and what you were doing when the problem occurred.
2. Any corrective action that you have tried.
3. The exact wording of any messages that appeared on the display.

Note:	Do not service 2020 in a hazardous location.

6.2. Fault Messages

When the "Falt" status is displayed, 2020's operation is compromised.

Falt1: Signal from zero gas is too high.

Cause: If another fault occurred while 2020 was setting its zero point, then this fault is displayed.

Action: Ensure no faults are occurring and calibrate 2020 again.

Cause: Contamination of sample line, sample probe or fittings before the detector.

Action: Clean or replace the sample line, sample probe or the inlet filter. See Section 5.3.

Cause: Span gas and zero air are mixed up.

Action: Ensure clean air is used to zero 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Cause: Ambient air is contaminated.

Action: If you are unsure about the quality of ambient air, use a supply of commercial zero grade air to zero 2020. See Section 3.1.3.

Falt2: Signal from span gas is too small.

Cause: Span gas and zero air mixed up.

Action: Ensure clean air is used to zero 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Action: Ensure the span gas is of a reliable concentration.

Cause: UV lamp window is dirty.

Note:	Do not remove the detector lamp in a hazardous location.

Action: Clean the UV lamp window. See Section 5.2.2

Cause: UV lamp is failing.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp. See Section 5.2.1.

Cause: Incompatible application.

Action: The concentration and sample gas are incompatible for use with 2020.

Falt3: UV lamp fault. UV lamp has not started.

Cause: UV lamp has not started immediately.

Action: This fault may be seen momentarily when 2020 is first turned on. Allow 30 to 60 seconds for the UV lamp to start and the fault to clear.

Cause: UV lamp serial number label is blocking the photocell.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the label is blocking the photocell. Rotate the lamp approximately 90° and then try to start 2020 again. If the fault persists, replace the lamp.

Cause: UV lamp not installed.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Action: Install a UV lamp. See Section 5.2.1.

Cause: UV lamp has failed.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp. See Section 5.2.1.

Cause: Electronic problem.

Action: If a new UV lamp still generates this fault, then contact the Photovac Service Department.

Falt4: Pump current too low or too high.

Cause: If the pump sounds labored, then the pump is operating beyond normal operating parameters.

Action: Check for an obstruction in the sample line. Make sure sample line, sample probe or inlet filter are not plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter. See Section 5.3.

Action: Ensure the sample outlet, located on the underside of 2020, is not obstructed.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact Photovac Service.

Cause: 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Photovac Service Department.

Cause: The pump has failed.

Action: Contact Photovac Service.

3. Troubleshooting

Problem: Very low or no instrument response detected, yet compounds are known to be present.

Cause: 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 3.2 or 3.3.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Photovac Service Department.

Note: Do not remove or recharge the battery pack in a hazardous location

Action: Disconnect the battery charger before calibrating 2020. Section 3.1 or 3.3 for calibration instructions.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory. See Section 3.4.

Cause: Response factor has been set to zero.

Action: Enter the correct response factor. Refer to Appendix 8.6 for a list of response factors. If the compound is not listed in Appendix 8.6 or you are measuring gas mixtures, then enter a value of 1.0.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application. See Section 3.2.2 or 3.3.2.

Note: It does not matter which Cal Memory is selected or which response factor is entered, 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector is leaking. A decrease in sensitivity may be due to a leak in the detector.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Ensure the UV lamp has been installed correctly. See Section 5.2.1.

Action: Ensure the lamp cover has been tightened down. Do not overtighten the cover.

Action: Ensure the o-ring seal on the lamp cover is positioned correctly. See Section 5.2.1.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact Photovac Service. See Section 5.2.1.

Cause: UV lamp is too wide, causing flow to be restricted.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact Photovac Service.

Cause: Sampling environment is extremely humid.

Action: Water vapor is not ionized by the PID, but it does scatter and absorb the light and results in a lower reading.

The 2020 detector has been designed to operate under high humidity conditions. Under extreme conditions you may notice decreased response due to humidity.

Cause: UV lamp is failing.

Note:	Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp. See Section 5.2.1.

Cause: High concentration of non-ionizable compounds.

Action: Chemical compounds, such as methane, with IPs greater than the 10.6 eV scatter and absorb the UV light. Sensitivity may be decreased significantly.

Application with high backgrounds of such materials, may be incompatible with 2020. Contact the Photovac Applications Group for more information.

Problem: Erroneously high readings.

Cause: Sampling environment is extremely humid.

Action: Water vapor may contain mineral salts which carry a charge. The water vapor becomes an electrolytic solution which becomes ionized when it enters the detector.

Atmospheric water in areas around the sea or stagnant water may produce a response in the absence of contaminants. The same effect may be seen when conducting ground water investigations in areas where the water is hard because it contains a significant concentration of minerals.

Cause: 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 3.1 or 3.3.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Photovac Service Department.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory. See Section 3.4.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application. See Section 3.2.2 or 3.3.2.

Note:	It does not matter which Cal Memory is selected or which response factor is entered, 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Photovac Service Department.

Problem: Date and time settings are not retained.

Cause: The battery pack has been removed before 2020 was turned off.

Note: Do not remove or recharge the battery pack in a hazardous location

Action: Replace the battery pack and reset the time and date. Ensure 2020 has been turned off before removing the battery pack. See Section 1.5.1.

Cause: 2020 has not been used for 3 months or more and the internal battery (not the external battery pack) has discharged.

Note: Do not remove or recharge the battery pack in a hazardous location

Action: Connect 2020 to the AC adapter and turn 2020 on. Turn the pump off. See Section 2.6.1. While 2020 is running the internal battery is charging. Leave the instrument running for approximately 24 hours.

Problem: Instrument status shows "Over".

Cause: High concentrations of gases and vapors will cause a rapid change in signal level. The detector and associated electronics may become temporarily saturated.

Action: Wait a few seconds for the status to return to normal. PIDs are designed to detect relatively low concentrations of gases and vapors. Exposure to very high concentrations may result in a very high or maximum response.

Cause: The detector has become saturated.

Action: Move 2020 to a location where it can sample clean air. Sample clean air until the reading stabilizes around 0.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Photovac Service Department.

Problem: Display is blank.

Cause: Battery pack is critically low.

Note: Do not remove or recharge the battery pack in a hazardous location

Action: Replace the battery pack or connect 2020 to the AC adapter.

Cause: The battery pack is not connected to the instrument correctly.

Action: Ensure the battery pack connector is securely attached to the connector on 2020. See Section 1.5.

Cause: There is an undetermined problem.

Action: Reset 2020. Turn the instrument on and disconnect the battery pack as outlined in Section 1.5.1. You must leave the instrument on while you disconnect the battery pack. This will reset the instrument. Reconnect the battery pack and close the battery hatch. Turn on 2020, set the time and date and program all the calibration memories that you are using.

Action: Contact the Photovac Service Department.

Problem: Sample flow rate is less than 300 ml/min.

Cause: Inlet filter is plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace inlet filter. See Section 5.3.

Cause: Inlet filter has not been installed properly.

Action: Ensure that the inlet filter has been installed correctly. See Section 5.3.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Photovac Service Department. See Section 5.2.1.

Cause: UV lamp is too wide, causing flow to be restricted.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Photovac Service Department.

Cause: 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Photovac Service Department.

Cause: Sample outlet is obstructed.

Action: Ensure the sample outlet in not obstructed in any way.

Cause: Pump has been damaged.

Action: Contact the Photovac Service Department.

Problem: Liquid has been aspirated.

Cause: 2020 has been exposed to a solvent that can pass through the inlet filter.

Action: Contact the Photovac Service Department.

Problem: Corrosive gases and vapors have been sampled.

Cause: 2020 has been exposed to corrosive gases and vapors.

Action: Corrosive gases and vapors can affect the electrodes within the detector as well as the lamp window. Prolonged exposure to corrosive materials may result in permanent fogging or etching of the window. If 2020 is exposed to corrosive material, contact the Photovac Service Department.

6.4. Printer Troubleshooting

Note: 2020 I/S is not classified for use in hazardous locations with a printer.

Problem: Printer will not print.

Cause: Printer is not connected properly.

Action: Make sure that you have the correct cable for your instrument. Ensure that the printer cable is properly connected. If you are using the serial to parallel converter ensure the cables are properly connected to the converter, to the instrument and to the printer.

Note: Turn the converter and 2020 off before connecting the cables.

Cause: Mechanical problem with the printer.

Action: Make sure that the printer is plugged in and turned on.

Action: Make sure that the printer is on-line, as shown on its control panel.

Action: Check the ribbon, the print wheel or cartridge, and the paper supply.

Cause: Printer cable is not configured correctly.

Action: In order to modify the existing printer cable, the pinout information for the printer connector is required. Refer to the printer user's manual for specific details.

The pinouts for the 2020 printer cable are shown in Figure 23. If modifying the cable is not possible, take the electrical and pinout information to a computer store where a suitable adapter may be obtained.

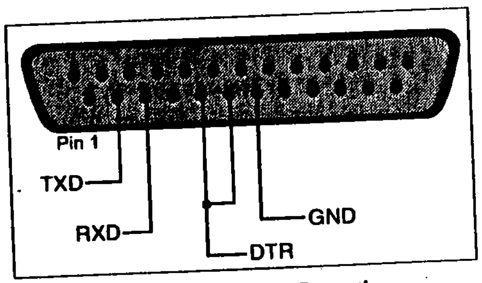

Figure 23 Printer Cable Configuration

Cause: Printer is not configured properly.

Action: Many printers have a set of configuration (DIP) switches. Generally, Photovac instruments expect these switches to be set in the factory default settings.

Many printers have switches for enabling automatic line feeds when receiving a carriage return. These switches should be set to carriage returns only, which is normally the factory default setting.

Some printers have switches or control panels that enable you to set the printer for different modes, such as sans serif, letter quality, or compressed text. Do not use these controls. If you do use them, you may cause your tabular or graphed output to be printed incorrectly.

Problem: Data are printed or downloaded correctly at first but become garbled.

Cause: Baud rate may be set too high.

Action: Ensure the baud rate of the printer is set to 9600 baud.

6.5. Serial to Parallel Converter Troubleshooting

Problem: Data are printed correctly at first but become garbled.

Cause: Baud rate may be set too high.

Action: Ensure the baud rate of the printer and the serial to parallel converter are set to 9600 baud.

Cause: Baud rate is not set correctly.

Action: Ensure the baud rate of the printer and the serial to parallel converter are set to the same value.

Problem: Printer will not print.

Cause: Problem with the printer.

Action. Refer to the corrective action in Section 6.4.

Cause: Serial card is installed in the printer.

Action: If a serial card is installed in your printer, you d not need the serial to parallel converter. Disconnect the converter and connect the instrument directly to the printer.

Cause: The serial to parallel converter is not turned on.

Action: Ensure the serial to parallel converter is connected to the AC adapter and the adapter is plugged into an AC outlet. The red LED on the top of the converter will be on. See Figure 16.

Cause: Cables are not configured correctly.

Action: You must use the correct type of cable to ensure trouble free data transmission. The length of the cable at the parallel side must not exceed 15 feet. On the serial side, use only the 2020 printer cable and suitable adapter.

When you are connecting the parallel side of the converter to a Centronics 36 pin, female connector, use a straight wired cable with 36 conductors and a Centronics 36 pin, male connector at each end. This cable is supplied with the converter.

When you are connecting the parallel side of the converter to a Centronics 36 pin, male connector, use a straight wired cable with 36 conductors and a Centronics 36 pin, female connector at one end and a Centronics 36 pin, male connector at the other end.

If you are connecting the parallel side of the converter to a female DB-25 pin connector use a straight wired cable with a DB-25 pin, male connector on one end and a 36 pin, Centronics, male connector on the other end. The pin assignments for all parallel port signals are listed in Table 8.

Pin #	Name	I/O Parallel to Serial	I/O Serial to Parallel
1	/Strobe	In	Out
2-9	Data0-7	In	Out
10	/Acknlg	Out	Not Used
11	Busy	Out	—
12	PE	Pull Low	Pull High
13	Select	Pull High	Pull Low
14	/Auto FF	Not Used	Not Used
15	NC	Not Used	Not Used
16	Ground	—	—
17	NC	Not Used	Not Used

Table 8 Serial to Parallel Converter Parallel Port Signals

Pin #	Name	I/O Parallel to Serial	I/O Serial to Parallel
18	NC	Not Used	Not Used
19-30	Ground	—	—
31	/Init	Not Used	Out
32	/Error	Pull High	Pull High
33	Ground	—	—
34	NC	Not Used	Not Used
35	NC	Not Used	Not Used
36	/Slct In	Not Used	Not Used

Table 8 Serial to Parallel Converter Parallel Port Signals - con't

The pin assignments for all serial port signals are listed below.

Pin #	Name	I/O Parallel to Serial	I/O Serial to Parallel
1	Frame Ground	—	—
2	TXD	Out	Out
3	RXD	In	In
6	DSR	In	—
7	GND	—	—
20	DTR	—	Out

Table 9 Serial to Parallel Converter Serial Port Signals

Note: A DB-25, female connector is sometimes a parallel port. Do not connect the serial port of the converter to a parallel port.

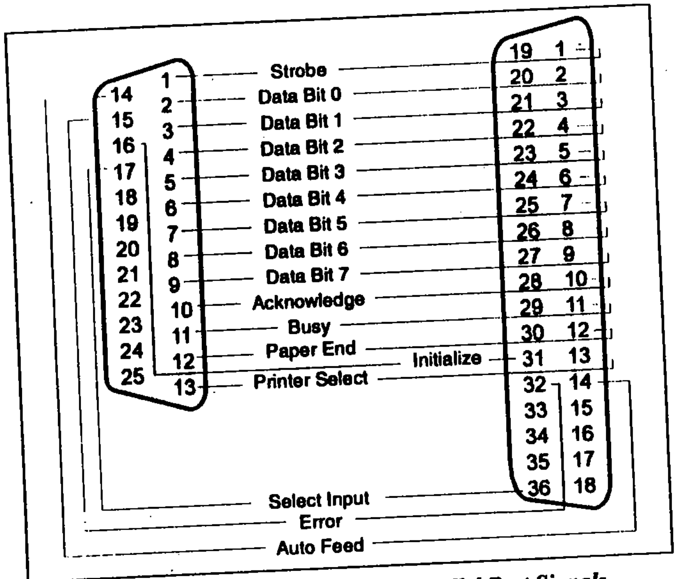

Figure 24 Serial to Parallel Converter Parallel Port Signals

6.6. Establishing Computer Communications

If, after having followed the procedure in Section 4.2, communications cannot be established with a computer, the problem may lie with the hardware connections or the printer cable configuration.

1. Ensure 2020 is connected to the serial port of the computer.

   The serial port will usually be a male connector, typically 9 pins on an IBM-AT® and compatibles and 25 pins on an IBM-XT® and compatibles. The 25 (or more) pin female connector is usually a parallel port. An exception to this rule is Tandy® Computers, which use a female 25 pin connector for the serial port. 2020 cannot be connected to a parallel port unless you are using the serial to parallel converter. (Photovac Part No. 380145)

   If you do not have a free serial port on you computer and you want to connect 2020 to the parallel port, you will need the serial to parallel converter. Connect 2020 to the serial port on the converter and then connect the computer to the parallel

port. If the port on the computer is anything other than a female, 36 pin Centronics connector you will need an adapter in order to use the cable supplied with the converter. See Section 6.5 for more details.

2. Ensure the cable being used is compatible with the device.

   An IBM-AT and compatibles with a 9 pin serial connector will first require a null modem to switch pins 2 and 3. Next a gender changer that converts the male DB-25 connector on the printer cable from male 25 pins to female 9 pins is required. These two adapters have been combined into one, which is supplied in the 2020 cable kit.

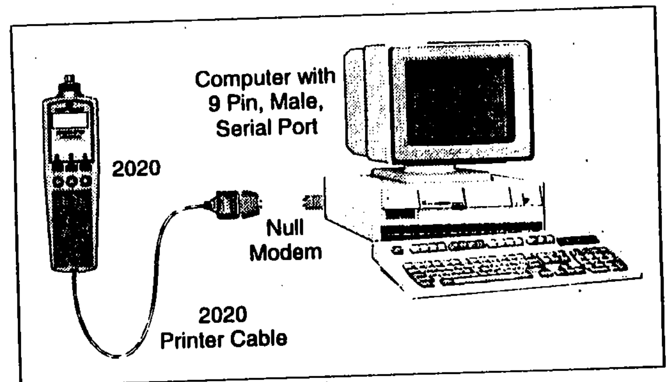

Figure 25 Connecting 2020 Using the Null Modem Cable

An IBM-XT and compatibles should not require a null modem but will require a gender changer. A gender changer will convert the male DB-25 connector on the printer cable to a female connector. A gender changer is supplied with the 2020 printer kit. See Figure 26.

The pin definitions of interest are listed in Table 11. Only pins 2 and 3 are shown since these are the problem pins:

Pin #	2020	IBM-AT	IBM-XT
2	RXD	RXD	TXD
3	TXD	TXD	RXD

Table 10 Serial Port Pin Definitions

Pins 2 and 3 should be mismatched between 2020 and the computer. Ensure this is the case. It is also possible that the cable being used may switch pins 2 and 3, even if it is not necessary. The cable may be a null modem. IBM-XT cables are usually null modems, since a null modem is required for connection to a printer.

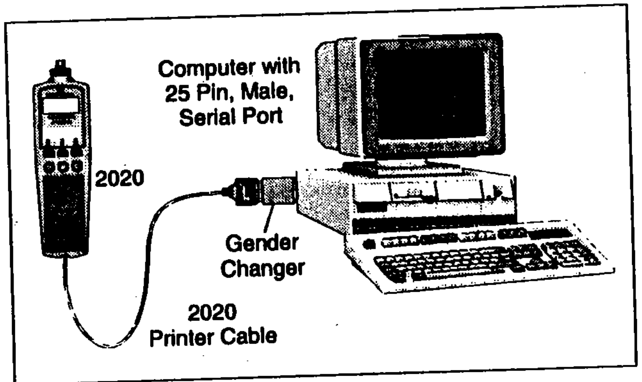

Figure 26 Connecting 2020 Using the Gender Changer

If you are using an IBM-AT and find that a null modem is not required it is possible that an IBM-XT serial port has been added to an expansion slot and thus does not require a null modem. The opposite may be the case if an IBM-AT serial port was added to an IBM-XT expansion slot, in which case the null modem is required.

3. Ensure all hardware is working properly.

   Use a printer to test both 2020 and the computer. Connect 2020 to the printer and ensure that this arrangement produces the desired results. If the correct printout is obtained, then the 2020 and the printer cable are okay.

   Now connect the computer to the printer and ensure this works. If the desired printout is obtained this ensures the computer parts are handling data correctly.

7. Technical Description

7.1. General Operation

2020 is a microprocessor controlled air monitor for measuring the presence of photoionizable chemicals in air at parts-per-million levels. The block diagram in Figure 27 shows the main components of 2020.

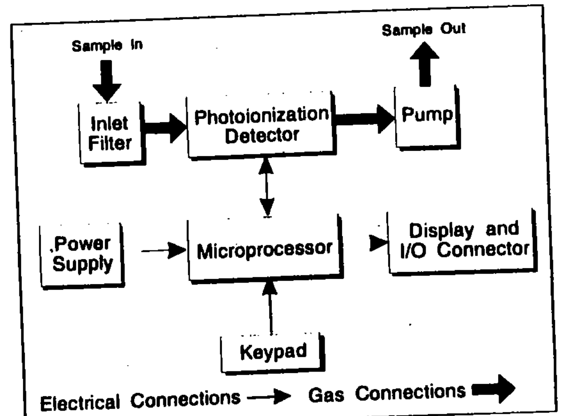

Figure 27 2020 Block Diagram

The microprocessor controls the components of the instrument and interprets and records the signal generated by the photoionization detector (PID). Recorded data and setup information entered into the microprocessor's memory are retained when 2020 is turned off.

A pump continuously pulls the air under test through 2020's PID. The PID converts the concentration of photoionizable chemicals in the sample into an electrical signal. The microprocessor subtracts any background from the signal and divides this signal by a sensitivity obtained by calibrating with a standard gas of known concentration. This concentration appears on 2020's display and, depending on the values entered through 2020's keypad, an alarm status may be displayed and an audio signal may be heard.

2020 can detect thousands of different types of airborne gases and vapors and its response depends on the type as well as the concentration. 2020 does not distinguish one type of chemical from another, but displays a number indicating the total concentration of all photoionizable compounds in the sample.

A standard of isobutylene at a known concentration may be used for setting the sensitivity. If 2020 is calibrated with isobutylene, it displays concentrations in units equivalent to ppm of isobutylene. If isobutylene were the only photoionizable chemical in the sample, then 2020 would display its concentration directly.

2020 responds more or less readily to other chemicals than it does to isobutylene. Because it has a medium sensitivity to isobutylene, this gas has been chosen as a reliable means of reporting an average concentration of total ionizables present.

For special applications, gases other than isobutylene can be used to calibrate 2020.

7.2. Photoionization Detector

2020's PID is shown in Figure 28. The PID measures the concentration of photoionizable chemicals in the gas stream from the sample inlet and produces an electrical signal for the microprocessor.

A UV lamp generates photons which ionize specific molecules in the gas stream. The permanent air gases (argon, carbon dioxide, nitrogen, oxygen, water vapor etc.) require a relatively high energy for ionization, and are not ionized by the UV photons. Many of the chemicals considered pollutants, including most hydrocarbons, are ionized.

The gas stream is directed into the PID through a small port at the center of the UV lamp window and through a series of larger ports around the perimeter of the lamp window. This arrangement permits a high sample flow rate and short response.

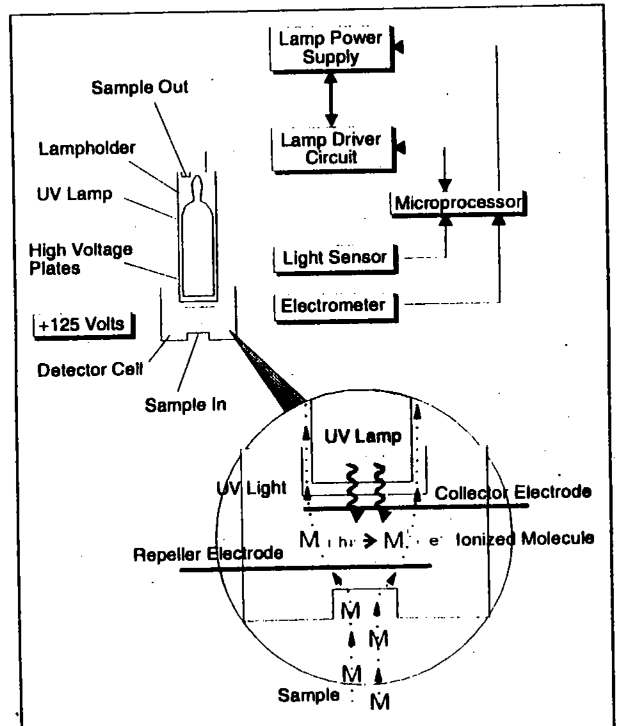

Figure 28 Photoionization Detector

The ionized molecules in the detector cell are subjected to a continuous electric field between the repeller electrode and the collector electrode. The ions move in the electric field, generating a current which is proportional to the concentration of the ionized molecules in the detector cell. An electrometer circuit converts the current to a voltage which is then fed to the microprocessor.

The detector lamp is operated by a high voltage lamp driver circuit which delivers high voltage energy to the lamp through plates in the lampholder. The lamp driver power supply is controlled by the microprocessor based on a feedback signal from a light sensor on the driver circuit board.

7.3. Calibration

Periodic calibration is required to compensate for 2020 output changes due to inlet filter restriction, ionization chamber cleanliness, pump wear and other factors.

During calibration, 2020 is first exposed to zero air. A small signal is generated. This zero signal is stored by the microprocessor.

2020 is next exposed to span gas. This span gas signal is stored by the microprocessor. The microprocessor subtracts the zero signal from the span gas signal and divides the difference by the user-entered span gas concentration. The resulting sensitivity is stored in the selected Cal Memory with the zero signal and the alarm levels. This number is then multiplied by the response factor and displayed.

2020 readings are always relative to the calibration gas. After calibration with isobutylene, 2020 will respond directly in units equivalent to isobutylene. Most volatile organic compounds will be detected by 2020. It cannot distinguish between isobutylene and other ionizable compounds. A reading of 10 ppm indicates all ionizable compounds that are present have generated an ion current proportional to 10 ppm of isobutylene. The reading is actually 10 ppm isobutylene equivalent units. 2020 readings give an indication of the total ionizables present and their concentration relative to the calibration gas.

7.4. Datalogger

7.4.1. Interval Operation

The microprocessor accumulates all readings in an averaging interval, that you select, and determines the minimum, average and maximum readings. It stores these numbers along with the highest priority instrument status and the most recent time and date. The datalogger can store 1000 of these entries.

These recorded data can now be reviewed and edited. Recorded data can also be printed as either a table or a graph. For each averaging interval, 2020 prints the minimum of all the minima, the average of all the readings for the interval and the maximum of all the maxima.

In PEAK mode, the reading is updated once a second. In the background, the 2020 datalogger is sampling the concentration and measuring min, max, and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the datalogger until the datalogger is full.

The MAX mode displays the maximum signal, with the date and time that it was recorded. 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is displayed on the meter display.

In STEL mode, 15 samples are combined to form a 15 minute average. Once every minute, the oldest of the 15 samples is replaced with a new one minute average. This moving average provides a 15 minute average with a one minute update rate so the meter display will only update once every minute. STEL is set to zero each time the instrument is turned on.

STEL calculations are always being performed by 2020. You can display the results of the calculations by selecting STEL as the Display mode

TWA mode sums concentrations every second until 8 hours of data have been accumulated. Once 8 hours of data have been summed, the accumulation stops.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While you are in TWA mode, the time on the status display will show the number of minutes and hours of data that TWA has accumulated. When this reaches 8 hours, 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by 2020. You can display the results of the calculations by selecting TWA as the Display mode.

7.4.2. Manual Mode

In manual operation you are prompted to locate a specific sampling site and then record both a background and a sample entry in the datalogger. 2020 stores these numbers along with the highest priority instrument status and the most recent time and date.

Recorded data can also be printed as either a table or a graph. The difference between the sample and background is calculated and shown on the printed output.

8. Appendices

3.1. Specifications

Size:	25.4 cm (10") long, 7.6 cm (3") wide, 5 cm (2") high
Weight:	0.8 kgs (1.75 lbs)
Detector:	Instant on photoionization detector.
Keypad:	6 silicone keys with tactile feedback
Status Display:	2-line, 16-character dot-matrix, backlit, liquid crystal display for alphanumeric readouts soft key display.
Meter Display:	4 digit, 7 segment display for real time concentration readout.
Datalogger memory:	16 kilobytes or 1000 entries
Serial output:	RS-232, 9600 baud, 8 data bits with no parity, for tabular and graphic printouts and connection to an IBM compatible computer
Audio output:	95 decibels @ 2048 Hz, on Alarm
Inlet connection:	1/8" compression fitting
Battery type:	Nickel cadmium rechargeable cell with intelligent charger

Charge/discharge time:	4 hr/8 hr
Battery charger:	Automatically charges and maintains full charge in battery pack
Materials in sample stream:	Stainless steel, Teflon®, Viton®, polypropylene, nitrile chorobutadiene rubber, nickel
Inlet filter:	Replaceable Teflon/Polypropylene, 1 um
Inlet flow rate:	Greater than 300 mL/min.
Operating temperature range:	0 to 40°C (32 to 105°F)
Operating humidity range:	0 to 100% relative humidity (non-condensing)
Operating concentration range:	0.5 to 2000 ppm, isobutylene
Accuracy:	+/-10% or +/-2 ppm, whichever is greater
Precision:	1% of calibration (calibrated with 100 ppm isobutylene)
Response time:	Less than 3 seconds to 90%
Detection limit:	0.5 ppm isobutylene
Dilution probe:	Normal calibration: concentrations between 100 to 20000 ppm +/- 20% High accuracy calibration: concentrations between 100 to 1000 ppm +/- 15%. Concentrations between 1000 to 20000 ppm +/- 20%

Note: Specifications subject to change without notice.

8.2. Warranty

2020 is warranted for one year against defects in materials and workmanship.

Photovac warrants that its manufactured product will be free from defects in materials and workmanship for a period of one (1) year from the date of receipt by the Customer. This may be voided if, in the opinion of Photovac, the product has been abused or treated in a negligent manner so as to cause damage or failure. Negligent use includes, but is not limited to, exposure of the internal parts of the equipment to water. Damage caused thereby is expressly excluded from this Warranty.

Consumable supplies and parts routinely replaced are not warranted.

Photovac and its vendors disclaim any implied warranty of merchantability or fitness for a particular purpose. Photovac and its vendors will not be liable for any indirect, special, incidental, or consequential damages, irrespective of whether Photovac or the vendor has advance notice of the possibility of such damages.

Photovac's sole liability under this warranty is limited to the repair or replacement of the product at its Service/Repair facility and return to the Customer.

When Photovac is made aware of a problem which would be eligible for remedy under Warranty, it will issue a Return Authorization Number to the Customer. No return will be accepted unless such authorization has been obtained. The customer is responsible for insurance and shipping to the designated Photovac Service/Repair facility.

In Canada:

Photovac Incorporated
330 Cochrane Drive
Markham, Ontario
Canada, L3R 8E5

Tel: (905)477-8088
Fax: (905)477-8220

In USA:

Photovac Monitoring Instruments
25-B Jefryn Boulevard West
Deer Park, New York
11729

Tel:(516)254-4330
Fax:(516)254-4284

In Europe:

Photovac Europa A/S
Sondervang 19
DK 4100 Ringsted
Denmark

Tel: +45-5767-5008
Fax: +45-5767-5018

In all other areas contact your Photovac representative.

8.3. Installing Alternate AC Plug on the Battery Charger

In most cases 2020 will be shipped with an AC line cord that will fit the AC wall outlet in your area. If this cannot be done, you may need to obtain an AC line cord suitable for the AC receptacle in your area.

The AC line cord, attached plug and receptacle must be marked with your country's certification mark and the cord must have a Harmonization (HAR) mark.

The line cord must be rated for either 100 to 120 VAC at 60 Hz or 220 to 240 VAC at 50 Hz. The voltage rating will depend on the voltage in your area.

Contact your Photovac representative to obtain more information.

8.4. Calibration Gas Supplier

The recommended span gas is isobutylene in air. 100 ppm isobutylene in air may be obtained from Photovac. (Photovac Part No. 350012 for Flow Match regulator, 395066 for gas bag calibration).

The exact concentration will be determined by your application. Other concentrations and other gases may be obtained from Scott Specialty Gases Inc. When ordering, specify a Scotty® V or Mini-Mix™ Cylinder.

Scott Specialty Gases Inc.
1290 Combermere Street
Troy, Michigan 48083

Telephone (within the USA): 1-800-774-9447
Telephone (outside USA): 1-810-589-2950
Fax: 810-589-2134

8.5. Using the Gas Bag

1. Turn the knurled plastic knob counterclockwise to unlock it. Use the knurled collar on the valve tube to gently push the valve tube down, toward the bag.

2. Turn the knurled plastic knob clockwise to lock the valve tube in place.

3. Turn the regulator knob counterclockwise about half a turn to start the flow of gas. Fill the gas bag about half full and then close the regulator.

4. Open the syringe port and empty the bag. Flush the bag a few times with the calibration gas and then fill it.

5. To close the gas bag valve, turn the knurled plastic knob counterclockwise to unlock it. Gently pull the valve tube up to close the valve. Turn the knurled plastic knob clockwise to tighten it against the valve tube.

Once the bag has been filled, use the bag and sample as soon as possible.

Note:	Do not use gas bags to sample unstable or highly reactive compounds. Do not use Tedlar® bags for storage of hazardous materials.

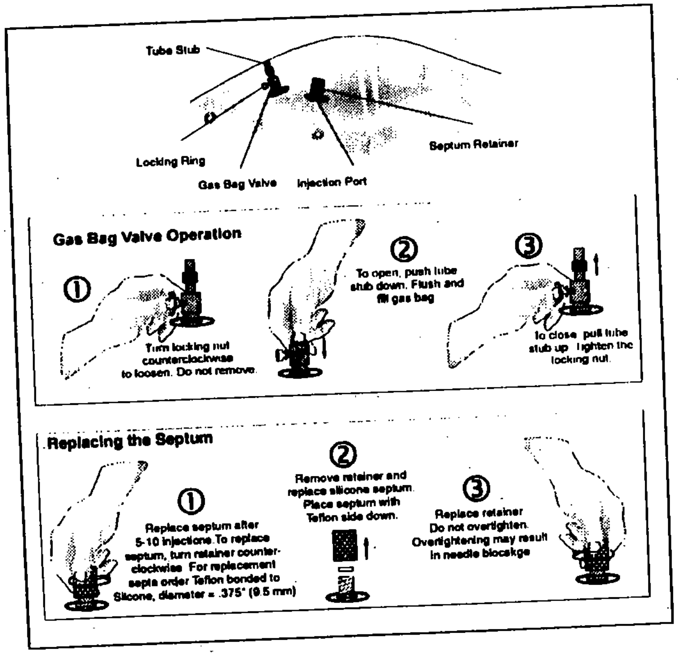

Figure 29 Using the Gas Bag

8.6. Response Factors

The response factors were determined over the range 5 - 500 ppm, based on a 100 ppm isobutylene calibration. isobutylene RF = 1.0. The following formula was used for calculation of response factors:

$$\text{Response Factor} = \frac{\text{Actual Concentration}}{\text{2020 Response}}$$

A response factor less than 1.0 indicates a compound response bette than that of isobutylene. A response factor greater than 1.0 indicates a lower response than that of isobutylene.

Note:	It does not matter which Cal Memory is selected or which response factor is entered, 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

When using response factors, results are expected to be accurate to +/- 10 ppm or +/- 25%, whichever is greater.

Compound	Response Factor
Acetaldehyde	10.5
Acetone	1.2
Acrolein (2-Propenal)	4.0
Allyl Chloride (3-Chloro-1-Propene)	3.9
Benzene	0.5
Bromoform (Tribromomethane)	2.0
1,3-Butadiene	0.7
n-Butanol	3.4
n-Butyl Acetate	2.3
n-Butyl Acrylate	1.8
n-Butyl Mercaptan (Butanethiol)	0.6
Carbon Disulfide	1.3
Chlorobenzene	0.4
Crotonaldehyde (2-Butenal)	1.2
Cumene (Isopropylbenzene)	0.6
Cyclohexane	1.3
Cyclohexanone	0.9
1,2-Dichlorobenzene (ortho-)	0.5
cis-1,2-Dichloroethylene	0.8

Table 11 Response Factors

Compound	Response Factor
trans-1,2-Dichloroethylene	0.4
N,N-Dimethylformamide (DMF)	0.8
1,4-Dioxane	1.3
Epichlorohydrin	6.5
Ethanol	8.8
Ethyl Acetate	3.8
Ethyl Acrylate	2.3
Ethylbenzene	0.5
Ethyl Cellosolve (2-Ethoxyethanol)	1.3
Ethyl Ether (Diethyl Ether)	1.2
Ethyl Mercaptan (Ethanethiol)	0.6
Ethylene	10.1
n-Heptane	2.4
n-Hexane	4.7
Hydrogen Sulfide	3.3
Isoamyl Acetate	1.8
Isobutyl Acetate	2.6
Isobutyraldehyde	1.1
Isopentane	8.2
Isoprene (2-Methyl-1,3-Butadiene)	0.6
Isopropanol	4.4
Isopropyl Acetate	2.6
Isopropyl Ether	0.8
Methyl Bromide (Bromomethane)	1.6
Methyl Ethyl Ketone (2-Butanone)	0.8
Methyl Isobutyl Ketone	1.0
Methyl Mercaptan	0.5
Methyl Methacrylate	1.4
Methyl tert-Butyl Ether (MTBE)	0.8
Monomethylamine	1.3
n-Nonane	1.4
iso-Octane (2,2,4-Trimethylpentane)	1.2
n-Pentane	10.4
n-Propanol	5.1
Propionaldehyde (Propanal)	14.8
n-Propyl Acetate	3.1
Propylene	1.2
Propylene Oxide	5.8

Table 11 Response Factors - continued

Compound	Response Factor
Styrene	0.4
Tetrachloroethylene (Perchloroethylene)	0.5
Tetrahydrofuran	1.5
Toluene	0.5
Trichloroethylene (TCE)	0.5
Trimethylamine	0.9
Vinyl Acetate	1.2
Vinyl Bromide	0.4
Vinyl Chloride (Chloroethylene)	1.7
Vinylidene Chloride (1,1-DCE)	0.8
meta -Xylene	0.5
ortho -Xylene	0.5
para-Xylene	0.5

Table 11 Response Factors - continued

Note:

1. Standards used for determination of response factors were from certified gas cylinders, +/- 2% analytical accuracy.
2. Response factors denoted with an asterisk (*) were determined over the range 25 - 250 ppm. These standards were prepared by addition of neat liquid to zero air.

8.7. Library Entries

Library selections simplify Cal Memory programming, and provide standard response factors and alarm levels for approximately 70 applications. The name, response factor and three alarm levels are all set from the library.

You can change any of the values entered in the Cal Memory. Changes, made to the library information that has been loaded into a Cal Memory, will have no effect on the original library entry.

Note: It does not matter which library or Cal Memory is selected or which response factor is entered, 2020's response is not specific to any one compound. The displayed reading represents the total concentration of all ionizable compounds in the sample.

See Section 2.6.4 for details on using libraries to program the Cal memories.

Compound	Code	RF
Acetaldehyde	ACETAL	10.5
Acetone	ACETONE	1.2
Acrolein (2-Propenal) 3	ACROLEIN	4.0
Allyl Chloride (3-Chloro-1-Propene) 3	ALLCHLOR	3.9
Benzene	BENZENE	0.5
Bromoform (Tribromomethane) 3	BROMFORM	2.0
1,3-Butadiene	13BUTADI	0.7
n-Butanol	nBUTANOL	3.4
n-Butyl Acetate	nBUTACET	2.3
n-Butyl Acrylate	nBUTACRY	1.8
n-Butyl Mercaptan (Butanethiol) 4	nBUTMERC	0.6
Carbon Disulfide	CS2	1.3
Chlorobenzene	CHLOBENZ	0.4
Crotonaldehyde (2-Butenal)	CROTONAL	1.2
Cumene (Isopropylbenzene)	CUMENE	0.6
Cyclohexane	CYCHEXAN	1.3
Cyclohexanone	CYCHEXON	0.9
1,2-Dichlorobenzene (ortho-)	12DCBENZ	0.5
cis-1,2-Dichloroethylene	cis12DCE	0.8
trans-1,2-Dichloroethylene	trn12DCE	0.4
N,N-Dimethylformamide (DMF)	N,N-DMF	0.8
1,4-Dioxane	DIOXANE	1.3
Epichlorohydrin 3	EPICLHYD	6.5
Ethanol	ETHANOL	8.8
Ethyl Acetate	ETHYACET	3.8
Ethyl Acrylate	ETHYACRY	2.3
Ethylbenzene	ETBENZEN	0.5
Ethyl Cellosolve (2-Ethoxyethanol)	ETHCELLO	1.3
Ethyl Ether (Diethyl Ether)	ETHETHER	1.2
Ethyl Mercaptan (Ethanethiol) 4	ETHMERC	0.6
Ethylene 5	ETHYLENE	10.1
n-Heptane	nHEPTANE	2.4
n-Hexane	nHEXANE	4.7

Table 12 Library Entries

Compound	Code	RF
Hydrogen Sulfide	H2S	3.[illegible]
Isoamyl Acetate	IAMYACET	1.8
Isobutyl Acetate	IBUTACET	2.6
Isobutyraldehyde 6	IBUTALDE	1.1
Isopentane	IPENTANE	8.2
Isoprene (2-Methyl-1,3-Butadiene) 6	ISOPRENE	0.[illegible]
Isopropanol	IPA	4.4
Isopropyl Acetate	IPACETAT	2.[illegible]
Isopropyl Ether	IPROPETH	0.[illegible]
Methyl Bromide (Bromomethane)	MeBROM	1.[illegible]
Methyl Ethyl Ketone	MEK	0.[illegible]
Methyl Isobutyl Ketone	MIBK	1.[illegible]
Methyl Mercaptan (Methanethiol) 4	METHMERC	0.[illegible]
Methyl Methacrylate	MeMeACRY	1.[illegible]
Methyl tert-Butyl Ether (MTBE)	MTBE	0.[illegible]
Monomethylamine	MMeAMINE	1.[illegible]
n-Nonane	nNONANE	1.[illegible]
iso-Octane (2,2,4-Trimethylpentane)	IOCTANE	1.[illegible]
n-Pentane	nPENTANE	10.[illegible]
n-Propanol	nPA	5.[illegible]
Propionaldehyde (Propanal) 3	PROPANAL	14.[illegible]
n-Propyl Acetate	nPROACET	3.[illegible]
Propylene 5	PROPYLEN	1.[illegible]
Propylene Oxide	PROPOXID	5.[illegible]
Styrene	STYRENE	0.[illegible]
Tetrachloroethylene (PCE)	PCE	0.[illegible]
Tetrahydrofuran	THF	1.[illegible]
Toluene	TOLUENE	0.[illegible]
Trichloroethylene	TCE	0.[illegible]
Trimethylamine	TRMeAMIN	0.[illegible]
Vinyl Acetate	VINACET	1.[illegible]
Vinyl Bromide	VINBROM	0.[illegible]
Vinyl Chloride (Chloroethylene)	VINCHLOR	1.[illegible]

Table 12 Library Entries - continued

Compound	Code	RF
Vinylidene Chloride (1,1-DCE)	1,1-DCE	**0.8**
meta-Xylene	mXYLENE	**0.5**
ortho-Xylene	oXYLENE	**0.5**
para-Xylene	pXYLENE	**0.5**

Table 12 Library Entries - continued

Notes

1 Peak alarm levels have been established as the TLV-Ceiling concentration, or the TLV-STEL concentration in those cases where no TLV-Ceiling value exists.

2 In cases where no STEL exists for a compound, the STEL value has been established as equivalent to the TWA value.

3 In cases where recommended exposure limits are below the detection limit for the compound in question, the estimated lower limit of detection has been substituted for those values.

4 2020 is not suitable for monitoring of these compounds at ACGIH recommended levels.

5 A 1000 ppm TWA has been established for those compounds which are "Simple Asphyxiants", and for which no exposure value exists.

6 For those compounds which lack established exposure levels, an arbitrary value of 200 ppm has been established for the TWA, STEL and PEAK alarms.

8.8. References

1. Maslansky, Carol J. and Steven P. Maslansky. *Air Monitoring Instrumentation.* New York: Van Nostrand Reinhold, 1993

2. American Conference of Governmental Industrial Hygienists. ***Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices (1994-1995).*** Reprinted with permission

Note: The TLV/BEI™ publication is revised annually.

3. Scott Specialty Gases, Catalog, 1994

4. National Institute for Occupational Safety and Health. *Recommendations for Occupational Safety and Health, Compendium of Policy Documents and Statements*, January 1992.

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APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES

APPENDIX C

TABLE OF CONTENTS

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1001

Title: **Elements of SOP and Method Formats**

Revision history:

Number	Date
4	5/3/99
3	7/22/94
2	9/16/87
1	7/31/86

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 5-3-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 5-6-99

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to define the process of creating SOPs and Methods.

1.2 Scope

1.2.1 SOPs are considered to be administrative and other non-analytical tasks.

1.2.2 Methods are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. Both have similarities and differences in structure and necessary elements. This document assumes that the user either has some knowledge of the word processor being used or can figure out how to perform the basic operations necessary.

1.2.3 This SOP does not address document control except as it relates to numbering of the SOP. Document control is addressed in Laucks SOP LTL-1002.

2. Procedures

2.1 Word Processing Format

2.1.1 All new SOPs are written in WORD 6 format (or higher when accepted by QA). Older versions of SOPs may be written in WORD 2 or XyWrite, however, these formats should be updated to meet the current format as they are revised.

2.1.2 Flow charts may either be photocopied from methods, scanned and inserted into the document electronically or prepared from programs such as Flow 4 (Patton & Patton Software) and inserted into the document electronically. Any of these electronic files should be kept together with the electronic version of the document to facilitate its modification and inclusion into future updates.

2.2 Initiating an SOP or Method

2.2.1 Prior to creating a new SOP or Method or revising an existing document, the prospective author or supervisor should first complete a Document Control Form as specified in the document control SOP, LTL-1002. This form can be obtained from the QA Department.

2.2.2 SOPs are considered to be administrative and other non-analytical tasks. Methods are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. For tracking and control purposes, all will be assigned a number by the QA Officer or designee. This number will begin with the letters LTL- (such as this

SOP, LTL-1001). A number should be obtained from QA before the author begins writing the SOP but if this is not possible, a number <u>must</u> be obtained before the SOP can be turned in for review.

2.2.3 If a revision of a previous document is being undertaken, the SOP or Method number will remain the same but the revision number will be incremented. Revision numbers of new documents will automatically be assigned as 0 with subsequent revisions generally being incremented by 1 (1, 2, 3, etc.).

2.2.4 The author may then use the appropriate <u>Word</u> template (SOPhead, Iorgtemp or Orgtemp) from the [File][New] menu presented in <u>Word</u>. Hardcopies of these formats are not included in this SOP but may be accessed by the reader using the above means. These formats may change somewhat without updating of this SOP but if the author accesses the template from the laboratory network in this manner, the latest version will be automatically used.

2.2.5 These are meant for guidance only and changes to the formats will be allowed if they present a more complete and accurate account of method performance. While it is entirely up to the author to change any part of one of these templates to suit the specific procedure, these elements will be looked at in the review process and must be included unless they are inappropriate to the procedure being described.

2.2.5.1 **<u>SOPhead</u>** is a general SOP template. This template is NOT to be used for analytical methods as it does not contain all of the necessary elements of a method (i.e. QC requirements). Specific elements of a method are outlined below and in the method templates Iorgtemp and Orgtemp.

2.2.5.2 **<u>Orgtemp</u>** is the method template which has been created for chromatographic analytical methods. It is primarily written for organic analysis but may also be applied to such inorganic techniques as ion chromatography.

2.2.5.3 **<u>Iorgtemp</u>** is for most non-chromatographic methods, which comprises most inorganic analyses.

2.2.6 The template should be opened and appropriate information filled in. Most of the items which should need input are highlighted in red in the template. This does not mean that text which is black cannot be modified or even deleted if it is not pertinent to the analysis in question.

2.2.7 Draft versions of SOPs should be worked on in the "projects" drive (p: on most computers). They should then be located in the p:\sop directory under the subdirectory most relevant to the areas addressed by the SOP. For instance, in writing this SOP, it was stored in p:\sop\qa_sops. Metals SOPs would be worked on in the p:\sop\metals subdirectory. QA will transfer the final document to the appropriate location for permanent storage and archival in order to maintain copies of all of the appropriate revisions.

2.3 Revising an SOP

2.3.1 QA will transfer the last revision of the SOP to P:\SOP\[*department*] where the author will make whatever changes are considered necessary. Note, the file will now be either a .doc file or given the extension .R00, .R01, .R02....(depending on the revision), rather than a blank template. As noted earlier, if the older version in not in the latest Word format, it should be converted. After acceptance of the revision, QA will again transfer the approved revision back to a generally inaccessible location in the QA directory.

3. Elements Of An SOP/Method

3.1 Elements

3.1.1 Almost all SOPs and methods are referred to in the general sense as SOPs. However, in some sense, they differ.

3.1.2 SOP formats are more general and free-form, not requiring the same specific elements as a Method. SOPs need only have the appropriate cover (title and revision number as on the cover of this SOP), header information, table of contents, introduction and scope, and specific operating procedures (including any appropriate appendices). Other elements may be present, depending upon the subject, but since SOPs will cover rather broad-ranging topics, no repetitive elements other than the above are currently considered necessary.

3.1.3 Methods contain the appropriate cover (title and revision number as on the cover of this SOP), header information, table of contents, introduction and scope, equipment, reagents, specific operating procedures, calibration and quality control (including corrective actions), and any appropriate appendices. They should also include data package assembly information and run sequences. Appendices should include preparation of standard solutions, a Method QC Table and a procedural flow chart.

3.1.4 All of the SOP/Method templates contain a title page. The title page consists of the following features:

- The laboratory name
- The SOP/Method number (assigned by QA Officer)
- The title of the SOP including EPA, SW846, Standard Methods or other method number reference when appropriate
- The revision history (revision number and date of approved revision)
- Signature of Author and date signed
- Signature of managerial reviewers (minimally, the QA Officer and Lab Director but may include the Divisional Manager and/or Technical Director)

3.1.5 All of the SOP/Method templates contain a header record which identifies the SOP/Method number, revision, date, page number and pages, and the method or revision it replaces (if any), such as for this SOP. The header should appear on all pages except the cover page and any pages that may be attached as appendices which are not part of the document itself. This record is not readily apparent in the "normal" mode of Word templates but must be completed by the author.

3.1.5.1 In Word, choose [View][Header/Footer]. Then fill in the appropriate information, and [Close]. This information may be modified later by following the same steps. It may also be modified by using the [Page Layout] selection from the [View] mode and changing the appropriate selection.

3.1.6 Though not required, it is preferred that the SOP also contain a footer which identifies the laboratory. It is preferred that both header and footer are separated from the document text by a double line followed (header) or prefaced (footer) by a carriage return, such as on this SOP to separate the header or footer from the text.

3.1.7 All of the SOP/Method templates contain a Table of Contents. The table of contents will be titled as such and include the header information. It should enumerate all of the major sections of the SOP and where they are located, including appendices.

3.1.8 All SOPs/Methods contain an Introduction and Scope. This section should include a brief description of the process delineated in the rest of the text. Where the process described varies from an accepted methodology (such as SW 846 or CLP), the variations should be clearly depicted in this section.

3.1.9 In methods, sample collection. storage, and holding times should be clearly outlined.

3.1.10 A part defining terms, particularly those which are specific to that procedure and may not be familiar to all readers is a valuable element of any procedure. This section is a standard part of the templates.

3.1.11 All SOPs/Methods contain a section called Equipment List and Standards (and/or reagents). All equipment and solutions necessary to complete the process described should be outlined in this section.

3.1.12 All SOPs/Methods contain a section called Safety Precautions and Waste Disposal. Any potential safety hazards should be depicted here as well as all waste disposal processes that may be entailed. If disposal involves pouring the waste into a collection container, that is all the description that is needed. The SOP then only need reference the waste disposal SOP for final disposal.

3.1.13 Where appropriate (almost always in Methods), the document should contain a section on Calibration and Quality Control. This will discuss all elements related to calibration and calibration verification. It will also discuss QC samples, frequency of all calibration and QC samples, criteria for all of these samples (including how to calculate %D, % recovery, RPD or whatever other criteria that might be appropriate), and corrective actions should any of them fail to meet their respective criteria. For most methods, a table should also be provided in one of the appendices which briefly outlines this same information. The bulk of the descriptive text, however, must appear in this section.

3.1.14 A section called Operation Procedures must be included in all SOPs/Methods which thoroughly describes the actual process. Some might consider this to be the heart of the procedure, where all analytical or other operational information is fully described in sufficient detail such that one who is reasonably familiar with the process could perform the procedure using only the SOP, with no special knowledge other than the basic principles involved and a general competence in the techniques.

3.1.15 A section should be contained in all methods called Reports. This should outline all analytical and QC reports and how they are presented, including control charts for many methods. This section should also include data package organization. If it is simpler to present some of this information in an appendix, the author may choose to use this approach. However, authors are encouraged to minimize the necessity of readers to reference too many sections of a procedure at one time to figure out all of the specifics of a process. In other words, it should be as easy to follow as possible and not force the reader to look in multiple sections of the SOP to find all of the information necessary for one relatively small part of the process.

3.1.16 Finally, SOPs and Methods, while not always required, will often contain Appendices. Two specific appendices common to most methods are a Quality Control Summary Table and a flow chart which depicts the basic steps involved in completing the process in the routine order and which includes the evaluation of successful completion of that process (i.e. "Is the QC in Control? If so, report the data. If not, what next?")

3.2 Saving the Document

3.2.1 Save the document under a name that will be readily recognizable. Generally, QA will store SOPs using the SOP number LTL-XXXX with extensions .r00, .r01, etc. to denote the revision. An update should be renamed by incrementing the extension by 1 (i.e. .r01 becomes .r02, etc.). It is not necessary that the writer of the SOP use this convention but may save it as a normal WORD .doc file with a readily recognizable name. QA will then rename the SOP when it is returned to permanent storage.

3.2.2 In Word, this may be accomplished by selecting [File][Save as] and filling in the requested information.

3.2.3 Note that if you want to save the document to any other drive or directory than it was called from, you will have to specify that path. The same conventions should be used to store the document as were discussed earlier in section 2.

3.3 After Completion of the Draft SOP

3.3.1 After the document has been written to the satisfaction of the author, it should be first passed to the department/division manager (unless that's who wrote it) for technical review. From there, the QA Officer and Lab Director will review and approve the document. QA will distribute all approved and signed documents. An unapproved (fully signed) SOP or Method document is not considered official. Other details of the document tracking process are discussed in that SOP (LTL-1002).

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1002

Title: **Document Tracking and Control**

Revision history:

Number	**Date**
1.0	05/15/95
2.0	12/27/95
3.0	1/06/98
4.0	12/14/99

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 12-14-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 12-15-99

Controlled Document

No. 20 Assigned to: TeTra

Table of Contents

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to describe the system under which Laucks creates and tracks controlled documents. This insures that the latest, approved version is in use and that prior versions are kept on file but are not available for unauthorized use. It forbids the use of unapproved or expired copies of methods or procedural documents. This includes but is not limited to procedural SOPs, QA documents, and analytical methods. Other documents may be included under this system at Laucks discretion.

1.2 Document Types

1.2.1 Laucks recognizes two types of documents.

- SOPs are considered to be administrative (such as this document or others dealing with data review or sample entry) or they may be analytical procedures (methods).
- Guidance and other miscellaneous documents may be generally broader in scope and utility than SOPs, examples being the laboratory QA Plan, Software Quality Assurance Plan or Chemical Hygiene Plan.

1.3 Scope

1.3.1 The protocol for initiating new documents is outlined, as well as the process for their approval. The tracking process is also outlined as is distribution to appropriate individuals and replacement of outdated copies with updated versions.

1.3.2 This SOP does not attempt to describe the actual creation of documents except to require that certain elements be present in order that the document may be tracked and controlled. Other SOPs (such as Elements of SOP and Method Formats) describe the structure or other elements required for a specific type of document.

2. Operation Procedures

2.1 Initiation and Updating of Documents

2.1.1 In order to track the status of documents, it is necessary to first be aware of what documents are in the process of being created, reviewed or revised. In order to do this, the Document Control Form is used (see Appendix A). Prior to beginning the creation or revision of any SOP or other controlled document, this form should be filled out. It will be kept on file in the QA Department so that it will be known which documents are in the process of being written or revised, and who is the primary responsible person for creating, reviewing or revising it.

2.1.2 The form should be filled out by either the individual responsible for the creation or revision, their Department Supervisor, or Division Manager. Creation or revision of documents may also be assigned by the Laboratory Director, Technical Director, or QA Officer to specific individuals. The form, however, must be approved and kept on file by the QA Department.

2.1.3 Copies of this form will be given to the responsible individual and the appropriate Division Manager. Originals will be kept on file in the QA Department. This insures that all responsible parties are informed of the initiation of the creation or revision process. This form should be filled out as soon as it is determined that the creation or revision of a document is necessary and a responsible party has been assigned. These forms will also be issued approximately annually in order to initiate the review process for existing SOPs.

2.1.4 It is recognized that some documents may have been written prior to completion of the Document Control Form or that it may be decided that some documents which are already in existence should be placed into the document control system. Unless these documents are ready for immediate approval, and acceptance by the Lab Director, QA Department and/or other responsible parties, in other words, not in a draft or review status, the document control form should be filled out.

2.1.5 Shortly after the Document Control Form is approved and distributed by the QA Department, an entry will be made in a database maintained by QA which tracks the status of that document. **All documents which have been previously approved but are currently in the process of being revised will remain in force until revisions have been completed and approved.**

2.2 Tracking and Control of Existing Documents

2.2.1 Most documents, particularly SOPs and administrative documents, will be assigned document numbers beginning with LTL. The scheme for numbering documents then proceeds as follows: The most important designator is the “thousands” place. If it is unclear which “hundreds” place designator is appropriate, the one which appears to be most appropriate may be used. This SOP will not be considered to have been violated if an incorrect “thousands” or “hundreds” place designator was used but every effort will be made to use the correct designators in order to maintain a more logical organization. This organization, although preferable, is not necessary for actual control of the documents as long as each complete LTL designator is unique. Unique numbering is enforced by the SOP database.

LTL-1000	QA / Administration
LTL-2000	Health and Safety
LTL-3000	Organic Extractions
LTL-4000	Sample Control,
-4100	Project Management
-4200	Document Management and Reporting
LTL-5000	Computer Systems (LIMS / MIS)
LTL-6000	Miscellaneous
LTL-7000	Metals Digestion
-7100	ICP Analyses
-7200	ICP/MS Analyses
-7300	Graphite Furnace Analyses
-7400	Flame Atomic Absorption Analyses
-7500	Cold Vapor Atomic Absorption Analyses
-7600	Gaseous Hydride Atomic Absorption Analyses
LTL-8000	Gas Chromatography, Volatiles
-8100	Gas Chromatography, Semivolatiles
-8200	GC / Mass Spectrometry
-8300	HPLC
-8400	Other Organic Analyses
LTL-9000	Conventional Chemistry- Titrimetric Analyses
-9100	Conventional Chemistry- Spectrophotometric / Instrumental Analyses
-9200	Conventional Chemistry- Gravimetric Analyses

2.2.2 Original documents will always be given a revision number of 0. Subsequent revisions, no matter how minor the revision, will be incremented by one.

2.2.3 In addition to the numbering and revision documentation, the document must also be given a title which will uniquely identify the document content. If the document is an analytical method, the method reference should be incorporated into the title. One example of this might be "Organochlorine Pesticides and PCBs by SW 846 Method 8081A."

2.2.4 SOPs, Methods, and many other documents must have header information which clearly indicates the document number, revision, date of revision, and document replaced by revision. Page numbers and the total number of pages in the document are strongly recommended and usually required, although some discretion may be allowed by QA in special circumstances. The header may vary in format but must contain all required information similar to the following. SOPs which have been created using the template described in the Laucks SOP on the Elements of SOP and Method Formats will contain the header information as described.

SOP No:	LTL-xxxx
Revision:	1
Date:	12/14/99
Page:	x of xx
Replaces:	0

2.2.5 As a minimum, approved documents are signed by the author, QA, and the Laboratory Director. They may also be signed by other critical supervisory personnel as deemed appropriate by QA. In general, methods will either be written by these supervisory personnel and not require an additional signature, or they will be written by an individual (signed), reviewed by a supervisor (signed), and approved by QA and the Lab Director.

2.2.6 Once a document has successfully undergone review and been signed-off by the author of the document and all of the other appropriate individuals (Laboratory Director, QA Officer, and, where appropriate, Technical Director, Division Managers, etc.), it is added to the SOP database list. Only approved documents and their most currently approved revisions are noted on these lists. These lists are broken down by department and distributed to department supervisors with the distribution date indicated. New lists are distributed whenever a new document or revision is added.

2.2.7 A database is maintained by the QA Department which, as a minimum, will track the document number, Department, revision number (or New or Draft if the document is incomplete), responsible individual, title and SOP Manual distribution (if the document has been completed and approved). Also tracked are the most recent revision date, the next revision due date, the last review date and, if any version existed under the previous SOP system, the previous SOP number. Only the applicable fields among these latter fields need be filled out. A copy of the screen form is presented in Appendix B.

2.2.8 Types of reports available from the SOP database include a table of contents for each SOP book, which are printed out whenever SOPs are released, automated Document Control Forms similar in content to the one in Appendix A, reports on SOPs due for review, and reports on SOPs overdue for review. The latter forms enable QA to assign and to track the status of SOPs which are up for their annual review. As this is an Access

Database, any other type of query or report form can be generated that uses any of the information previously noted in the above paragraph and in Appendix B.

2.2.9 Copies of the most current documents are kept on file in the QA Department and departmental specific documents are kept by the departmental supervisor in ring-binders which are available to all analysts and other appropriate staff. The SOP manuals are maintained in key locations throughout the laboratory. The SOP manuals contain only those SOPs pertinent to that area of the laboratory. The SOP manual locations are presented in Appendix C. These departmental copies are stamped with a Controlled Document Stamp (See Appendix D) in either red or black annotated with red pen. These copies, which are tracked by the QA department, will be replaced when a newer version has been completed and signed-off. The color of the Controlled Document Stamp and/or annotation, will be black on subsequent secondary copies and will not be directly tracked by the QA department as these documents are considered uncontrolled.

2.2.10 It is the **Departmental Supervisor's** responsibility to ensure that their staff have copies of the most recent version of any document available to them. Keeping copies of outdated versions is inappropriate as they may be inadvertently used by uninformed individuals. When revised versions are issued, the old versions will be collected from the SOP books and usually disposed. In addition, the SOP book table of contents will be updated to reflect the revised SOP(s).

2.2.11 It is inappropriate for any individual to be working from an unapproved copy of a method or procedure. This means that individuals must not be working from copies of controlled documents. If an individual must consult an SOP, they must consult a controlled copy, which is readily available in a number of areas throughout the laboratory.

2.2.12 When documents are distributed to the departmental supervisor, a copy of the signature list(s) for the specific document(s) is/are also distributed. The signature lists are returned to the QA department when completed.

2.2.13 Departmental supervisors will ensure that the most recent versions of all appropriate documents are made available to all affected staff members. When this occurs, three things must happen.

- Newly distributed versions are placed in the SOP manuals.
- The signature lists for the current documents are signed and dated as staff complete reading the SOPs. In addition, as staff new to a particular task (SOP) are trained, the departmental supervisor will ensure that they have read and signed the signature list

for that SOP. This may require that the supervisor request a new SOP signature list for that staff member so that they can sign the SOP for newly assigned tasks.

- The departmental supervisor is responsible for ensuring that all outdated versions of SOPs are discarded or destroyed when the newer revisions are issued.

2.2.14 Note that although any person capable of performing a documented task should be in possession of or have access to a current, officially assigned copy, the possession of a copy of any SOP or method does not imply that the individual in possession is qualified to perform the task detailed. They must still be properly trained in the techniques involved.

2.2.15 Note that versions of methods or SOPs which have been given to regulatory agencies or clients are generally uncontrolled in that they will not be updated except by specific arrangement. Controlled documents may be released to clients upon specific arrangement. However, the laboratory can only control the document and any updates up to the point they are provided to the client. It becomes the client's responsibility to then ensure that they are referencing the most recent versions in their own documentation.

2.3 Storage and Filing of Controlled Documents

2.3.1 Controlled documents will be kept by the QA Department. Master originals of the documents will be stored in a secure file and will generally not be used except to act as the reference copy and make intermediate "reproduction" copies.

2.3.2 Reproduction copies will be used to make subsequent copies for distribution to the laboratory and other authorities. These will be filed in QA but may not be stored in the same secure manner as the master copies.

2.3.3 Both master original and reproduction copies will be filed in order of their SOP number as defined previously.

2.3.4 Electronic versions of all controlled documents are also kept on file by QA. These versions are stored in an area of the laboratory network which has limited access to designated individuals. These electronic copies will be given names as closely matched as possible to their document or SOP number. Original documents and revisions will be given the extension .R00 or .R01, etc. to indicate their revision number. Should multiple files be necessary to create a given document, they will be incorporated into a subdirectory with similar naming conventions.

2.3.5 Copies of these electronic versions of SOPs will be distributed to individuals who have been assigned a revision. No other copies of these controlled documents should be kept by laboratory staff in order that unapproved copies of the document do not proliferate.

2.4 Review and Updating of Documents

2.4.1 In order to facilitate updates to documents without violating the practices outlined in the SOP, and in order to insure all approved updates have indeed been incorporated into the document, an "SOP Update" form (Appendix E) should be used. This form may be filled out at any time by an analyst or supervisor. Before the change can be brought into practice, however, it must approved by QA. QA may also choose to consult the area supervisor, Division Manager, or other senior staff before incorporating the procedure into the routine practice. A copy of this form will be kept with the laboratory controlled copy **AND** a copy must be filed with QA. When it is time to update the SOP, changes outlined on these forms will be incorporated into the revision.

2.4.2 Unless major changes to SOPs are required, SOPs should be reviewed approximately annually. Changes which do not require immediate update are typos or wording changes which do not inhibit the correct interpretation of the operation involved. Items which could lead to misinterpretation or incorrect performance of the methods should be corrected as soon as feasible. At the time of any revision, items addressed in the "SOP Update" forms will be incorporated into the SOP. In addition, any other updates determined at the time of the review will be added. Each review will be documented on the Document Control Form (Appendix A).

Appendix A

Document Control Form

Laucks Testing Laboratories

DOCUMENT CONTROL FORM

______ Generate new document

______ Modify existing document

______ Review existing document

Document No.: ________________

Document Title: ____

____

____

Assigned to: ________________________________ Date: ____________________

The aforementioned document has been reviewed and does not require modification at this time:

Reviewer: ________________________________ Date: ____________________

Purpose for generation or modification of document and comments on review:

____

____

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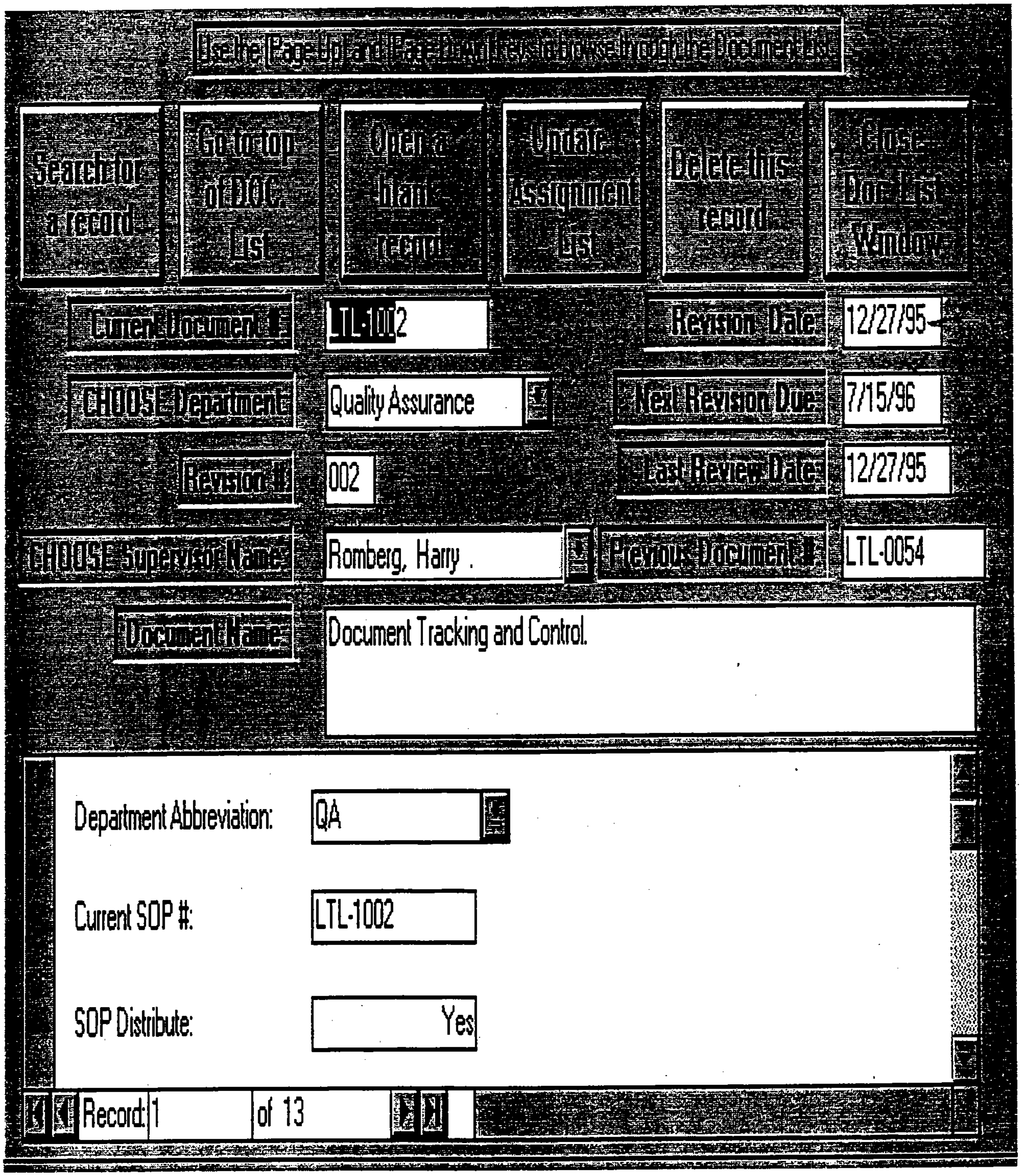
QA Approval: ________________________________ Date ____________________

Appendix B

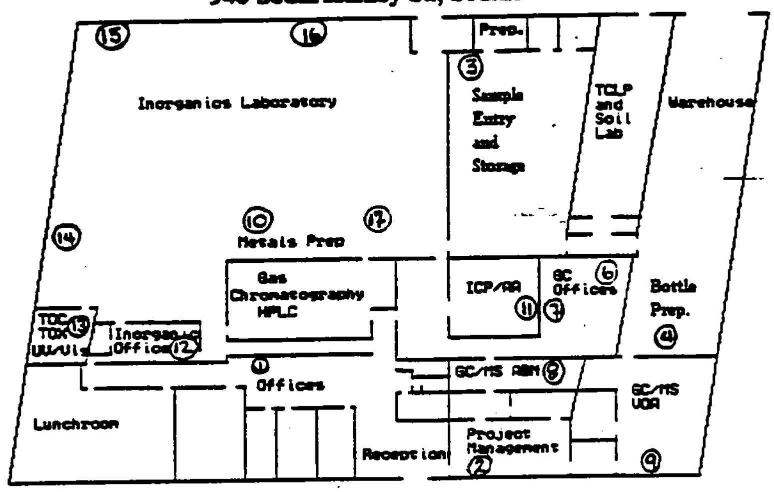
SOP Database Screen


Search for a record
Go to top of DOC List
Open a blank record
Update Assignment List
Delete this record
Close Doc List Window
Current Document #
LTL-1002
Revision Date
12/27/95
CHOOSE Department
Quality Assurance
Next Revision Due
7/15/96
Revision #
002
Last Review Date
12/27/95
CHOOSE Supervisor Name
Romberg, Harry .
Previous Document #
LTL-0054
Document Name
Document Tracking and Control.
Department Abbreviation:
QA
Current SOP #:
LTL-1002
SOP Distribute:
Yes
Record: 1
of 13

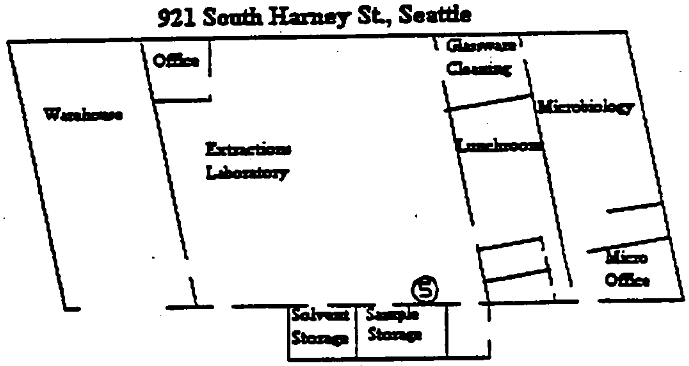
Appendix C

SOP Manual Distribution Locations

Laucks Testing Laboratories, Inc.
940 South Harney St., Seattle



Laucks Testing Laboratories, Inc.
921 South Harney St., Seattle



SOP Book Location

Appendix D

Document Control Stamp

Controlled Document

No. ______ Assigned to: ____________________

Appendix E

SOP Update Form

Laucks Testing Laboratories

SOP UPDATE FORM

Document No.: ____________________

Document Title:____

____

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The following changes have been reviewed and determined to be necessary to the implementation of the above document.

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Submitted by:____ Date:____________________________

Approved by (QA):____________________________________ Date:____________________________

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1003

Title: Chain-of-Custody and Documentation Procedures

Revision history:

Number	Date
1.	2/13/95
2	2/3/98

Written by: Harry Romberg
Harry Romberg, Quality Assurance Officer

Date: 2-3-98

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director

Date: 2/3/98

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1 Description

1.1.1 This SOP is intended to describe the chain-of-custody process at Laucks. for all samples from the point of receipt until the time of sample disposal. It does not address actual sample receipt, entry and log-in, nor does it address any aspect of samples analysis or reporting of results except as it pertains to maintaining the chain-of-custody. The chain-of-custody process is described only for samples requiring secure storage and strict chain-of-custody documentation.

1.1.2 The location of all samples requiring secure storage must be known at all times over the course of their possession by Laucks. Failure to maintain these conditions may result in invalidation of data on legal grounds, regardless of the technical level of data quality.

1.1.3 This process is restricted to use by, or under the supervision of analysts experienced in the process described. Each analyst or other individual requiring possession of the samples for any reason must understand the necessity of this documentation chain and be familiar with the process. Any person requiring access to the samples outside of the secure storage area must check them out using the described procedures.

1.1.4 Virtually all analytical staff and many others employed by Laucks are considered authorized personnel and may have access to one or more of the secure storage areas as needed for performance of their duties, at the discretion of the individual, and depending upon the nature of their duties. Removing of the samples or any aliquots thereof from the secure areas, however, requires completing the forms provided for this purpose. Individuals who are not Laucks employees will not have access to samples except under the direct observation and accompaniment of staff members.

1.2 Definition of Terms

1.2.1 Custody - A sample is considered under custody if:

- It is in the possession of an authorized person
- It is in view after being in the possession of an authorized individual
- It was in the possession of an authorized individual who then locked it up
- It is in a designated secure area which is accessible only to authorized personnel.

1.2.2 Chain of Custody - The process by which custody of a sample is maintained and documented throughout the period that the sample is in the possession of the laboratory.

Any changes in the possession (custody) of the sample must be documented in order that the chain-of-custody can be properly maintained.

2. Equipment List

- Secure Storage Custody Log(s), see Appendix A
- Volatiles Custody Log(s), see Appendix B

3. Safety Precautions

3.1 Safety Precautions

3.1.1 No safety precautions are necessary for adherence to the items addressed by this SOP. However, in handling actual samples while operating under this document, all standards, samples and sample solutions should be handled as if they are hazardous substances.

4. Operation procedures

4.1 Identification of Samples Requiring Strict Chain-Of-Custody

4.1.1 Almost all samples entering the laboratory come with chain-of-custody logs, either generated by the client or by Laucks. Often these chains-of-custody are intended only for clear identification of testing parameters, rather than actual custody maintenance. These custody logs, however, will always be signed, timed and dated by the person checking the samples in and entering them into the laboratory database.

4.1.2 Actual internal chain-of-custody procedures will be followed for all project and other work which require such procedures. These are usually identified as CLP work or work which require similar deliverables. These samples will usually, although not always, arrive with custody seals on the coolers and sometimes even the sample containers themselves. All work under the HAZWRAP, NFESC, or Army Corps of Engineers require these procedures, regardless of the type of deliverables requirements, as does any work involving pending legal action. If it is uncertain whether or not strict chain-of-custody should be maintained, these procedures should be followed.

4.2 Initiating Internal Chain-Of-Custody

4.2.1 Internal chain-of custody procedures begin when the samples are logged into the laboratory database. When the samples are logged into the system, they are stored in or near the sample entry area, in the main laboratory, in one of 3 locations:

- The main walk-in cooler is for organic extractables which have not yet been transferred to the extractions laboratory and for inorganics which require refrigeration.
- The volatiles refrigerators are located in the area between the GC room and the laboratory computer system hub.
- The locked "cage" in the log-in area is for samples not requiring refrigeration.

4.2.2 Additionally, samples requiring secure storage which are located in the walk-in will be on designated shelves. Those awaiting transferal to the organics extractions laboratory will be on their own designated shelf.

4.2.3 All of these areas are secured under lock and key, the keys being in the possession of sample control and, in the case of the volatiles refrigerators, in the possession of key analysts in those areas.

4.2.4 Samples requiring secure storage are logged into any of these areas by the sample receiving representative using a Secure Storage Custody Log (Appendix A). Samples not requiring secure storage need not have this form completed. A custody log will be completed for each **workorder** for which samples require chain-of-custody procedures.

4.3 Maintaining Internal Chain-Of-Custody

4.3.1 When samples are logged out of storage areas, they will be signed out in the appropriate spaces by the person removing them.

- If they are being removed for analysis, the "**Action**" column should state the analyses being performed. When they are returned, the logsheet must also indicate such. Additionally, the "**Sample Numbers**" column should indicate which samples are being removed for analysis (i.e. 1-10 metals digestion, or 3-5 NO3/NO2 analysis).
- If they are being removed for transferal to another location (i.e. extractions), the "**Action**" column should state where they are being transferred. Additionally, the "**Sample Numbers**" column should indicate which samples are being transferred (i.e. 1-10 volatiles, or 3-5 extractables).
- When samples are removed for final disposal, if all samples are being removed, the logsheet is signed and dated at the bottom of the page. If only certain samples are being disposed or to be even more clear, the "**Action**" column should indicate "disposed" and the "**Sample Numbers**" column should indicate which samples are being disposed.

4.3.2 When samples are signed into another storage location, this is done using an identical Secure Storage Custody Log. Samples which are subsequently removed from these areas for analysis or disposal should be signed out using the same procedures as above.

4.3.3 Volatiles samples, being generally for a single analysis, are signed out using an abbreviated logsheet. Copies of GC/MS and GC VOA logsheets are located in Appendix B.

4.3.4 Any analyst removing samples from **any** secure storage area for the purpose of preparation or analysis or transferal to another department **must** sign the samples out using the Secure Storage Custody Log and must sign the samples back in when they are returned, or must sign them into another secure storage area. Samples must be in the possession of the analyst who signed them out at all times during this period and must not be left unattended. If samples are analyzed and then immediately disposed, as may be the case for some volatiles analyses, the "**Action**" column on the custody log should indicate "analysis and disposal." Note, in checking volatiles samples out using a volatiles custody log, it is assumed that the purpose is to analyze for volatiles since no other analysis is performed on these samples.

4.3.5 There is ample room on any one Secure Storage Custody Log in almost all cases to accommodate checking all sample containers for a particular SDG or workorder in and out as often as required for all of the pertinent analyses from that secure area. Should an additional page be required, it should be obtained from Sample Entry. A second (or third) page must not be initiated until all of the space on the previous form has been filled. The first page must be marked "1 of 2" in the upper right corner and the second page marked "2 of 2". In the unlikely event that even more pages are required, this mark can be crossed out (single line, initialed and dated) and the sheets marked "1, 2, or 3 of 3, etc.

4.4 Sample Disposal and Closing of the Internal Chain-Of-Custody

4.4.1 When samples have been signed out for final disposal the chain-of-custody process is considered to be complete. The Secure Storage Custody Logs must be collated, bound and turned in to the Quality Assurance Department in order that the chain-of-custody can be tracked for all samples requiring this process, should such tracking be required at a later date.

4.5 Review of Custody Logs

4.5.1 On at least a quarterly basis, supervisory personnel must review selected custody logs to insure they are being filled out properly and completely. The supervisor need not review all of the forms but must review at least 5% or 10 forms, whichever is greater. The supervisor will stamp the reviewed forms with a "reviewed by" stamp , initialing and dating the notation or may write "reviewed by" by hand, likewise initialing and dating the form. The supervisor should also cursorily review the remaining forms to make sure there are no blatantly obvious omissions but need not mark these forms unless a discrepancy is observed.

4.5.2 Review should include noting that all spaces are filled in properly and completely (this especially means that all samples that were checked out must be checked back in and that the person handling the samples must be identified), that errors are crossed out with a single line, initialed and dated, that entries are clear and not obliterated.

4.5.3 Should the supervisor find errors or omissions, the must issue a corrective action form to the individual who made the error (if it is obvious who that is) or to the appropriate supervisor. Any of these individuals can correct the form (initialing and dating the correction). The primary purpose of the corrective action notice is so that the individual in error be re-trained as to the proper custody procedure.

4.5.4 Supervisors responsible for this review may assign the responsibility to other capable individuals but the ultimate responsibility is theirs. These supervisors are the Organics Division Manager for the volatiles logsheets, the Extractions supervisor for the extractables, and the Sample Control supervisor for the main lab walk-in and unrefrigerated secure storage areas.

Appendix A

Secure Storage Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:______________________________ **LTL Number:**____________________

Number of Containers (*optional*):____________________

Storage Unit:____________________ **SDG Number** (*optional*):____________________

Sample Number	Matrix (*optional*)	Location (*shelf*)	Logged Out Date	Logged Out Time	By	Logged In Date	Logged In Time	By	Action

Samples Disposed of by ____________________________ on ____________________

Appendix B

Volatiles Custody Logs

LAUCKS TESTING LABORATORIES
GC/MS VOA CUSTODY LOG

LABORATORY NUMBER	SAMPLE NUMBER	DATE & TIME REMOVED	BY	DATE & TIME RETURNED	BY	COMMENTS

GC Volatiles Custody Log
Laucks Testing Laboratories, Seattle, Washington

Samples	Analysis	Date/Time Removed	Analyst	Date/Time Returned	Analyst	Comment

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

Title: **Documentation of Analyst Competence and Training**

Revision history:

Number	Date
1.0	01/31/95
2.0	12/27/95
3	6/23/96
4	8/4/99

Revised by: Harry Romberg Date: 8-4-99
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 8-4-99
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope

1.1 Description

1.1.1 This SOP describes the way in which analyst competence is initially documented and by which the analyst is considered capable to perform independent analysis. Two practices are in place at the time of this writing. One practice is designed primarily for analysts who have been employed doing an analysis for a significant period of time at Laucks and have demonstrated competence through the successful analysis of many samples, including one or more of the following: performance evaluation (PE) samples, reference materials, laboratory control samples, surrogates, etc. The other practice is primarily for analysts who have been performing a specific analysis for less time than is considered extended proof of competence. This practice involves the analysis of multiple aliquots of a PE sample and subsequent evaluation of the results. *This practice also usually includes the completion of training checklists for the task for which the analyst is being trained.*

1.2 Scope

1.2.1 This SOP contains discussion of initial demonstration of competence through PE analysis and, for some analyses, P&A criteria. It also defines ongoing performance demonstration through the use of PE samples.

1.2.2 Specific elements of training in safety, QA, and in each department are maintained in separate files. However, quizzes and sign-off sheets from this training are included in the respective analyst's file as demonstration that such training occurred. Specifics of these types of training are not within the scope of this SOP.

2. Definitions

- **PE** - Performance Evaluation
- **P&A** - Precision and Accuracy
- **Trainer** - An individual who has documentation demonstrating experience recognition or successful completion of competency and has been performing the task/method for a minimum of 3 months experience for login, sample preparation, and reporting and a minimum of 6 months for analytical instrumentation operation and analysis reporting.

3. Responsibilities

3.1 Analyst

3.1.1 It is the responsibility of the analyst to complete all of the items of their required training in an appropriate timeframe as required by their manager, safety and QA.

3.1.2 The analyst must complete all demonstration of competency items outlined in this SOP in a manner consistent with the analytical SOP.

3.1.3 The analyst must analyze a PE study initially and on an ongoing basis (at least annually) for each method for which they are considered qualified.

3.1.4 For many analyses, the analyst must perform an initial Precision and Accuracy study as required.

3.1.5 The analyst must regularly perform all required method QC, including matrix and blank spikes and laboratory control sample analyses, which may be used to qualify them for competency.

3.2 Supervisor

3.2.1 It is the supervisor's responsibility to ensure that their analysts are all initially qualified to perform an analysis including ensuring that they have analyzed all required PE samples and performed all required P&A studies for the methods for which they will be doing analyses.

3.2.2 It is the supervisors responsibility to ensure that all analysts have participated in applicable QA and safety training.

3.2.3 It is the supervisor's s responsibility to ensure that on a continuing basis, at least annually, that analysts who are to be considered capable of performing an analysis, have performed within limits on at least one PE study for analyses for which such are available.

3.2.4 It is the supervisor's responsibility to ensure that other training has occurred, whether that means peer training, reading, quizzes, completed checklists, etc.

3.2.5 It is the supervisor's responsibility to develop and maintain current departmental training materials, such as checklists, quizzes, etc.

3.2.6 It is the supervisor's responsibility to ensure that the analyst's training file has been updated with the most current PE or P&A data as well as any quizzes or checklists that are considered part of their departmental training.

3.2.7 It is the supervisors responsibility to designate a qualified individual(s) to train personnel for their new task/assignment.

3.3 QA

3.3.1 QA maintains training files (except for Extractions where the supervisor maintains the files due to the location of the extractions facility).

3.3.2 QA periodically audits training files to ensure appropriate training is being maintained.

3.3.3 QA reviews PE and P&A studies to ensure criteria have been met.

3.3.4 QA works with managers to assist in developing training materials.

3.3.5 QA provides training to staff in QA issues and ensures that documentation of this training is in the staff training file.

3.4 Trainer

3.4.1 Completes applicable staff training documents during the training process.

3.4.2 Reviews documentation with the individual and the supervisor to ensure timely and accurate review of progress and documentation.

4. Operation procedures

4.1 Recognition of Experience and Training

4.1.1 Many analysts have been performing their assigned duties for an extended period of time and have successfully analyzed many samples, reference materials, PE samples, matrix, blank, and surrogate spikes and have not only demonstrated their capabilities to achieve results which meet criteria but have demonstrated a thorough knowledge of all aspects of the chemistry involved, instrument performance and maintenance, the necessary data reduction requirements, quality control criteria, and documentation.

4.1.2 These analysts, at the discretion of the appropriate Division Manger, may be certified to independently perform their analytical duties. This is achieved using the Recognition of Experience and Training Form, an example of which is in Appendix A. This form contains space to note the analysis type (Cyanide, for example) and the methods by which they are considered competent (335.3 and 9012 perhaps, but not CLP). The dates from which they have been doing these analyses must also be noted on the form. The Division Manager then signs the form in order to certify that the analyst is considered adequately trained in the particular method or aspect of the job. The form must include the criteria used to designate someone as competent and attached to the form must be the applicable documentation to confirm the criteria have been met.

4.1.3 Certification of competency must include the successful analysis of a performance evaluation (PE) sample where such are available or can be made in the laboratory by a supervisor. This sample will be blind to the analyst, must be analyzed independently by them and must be analyzed in accordance with the appropriate SOP. Greater specifics on these types of samples are given in the Laucks SOP entitled "Blind Spike Program" but will often be from a WP or WS study or from another commercial source. Analysts who have been performing analyses for any length of time at Laucks have almost certainly analyzed numerous PE samples which can be used for initial and ongoing demonstration of competency.

4.1.3.1 Adequate performance on a PE sample will be considered to be within the supplied statistical limits for that sample if from a commercial source or from method defined limits for an LCS or blank spike if from internally prepared material.

4.1.4 Precision and Accuracy (P&A) criteria using quadruplicate analysis are also a part of most organic SW846 and some other methods. Successful analysis of such samples will be considered to be within the reference method-specified criteria. Since Laucks own precision and accuracy limits must be within the method-specified criteria, the analyst should also be able to meet Laucks criteria as well as those of the reference method. However, as long as method criteria are met, the analyst may be approved for independent work as long as they are able to obtain satisfactory performance from the ongoing analytical QC for that analysis.

4.1.5 Competency may also be demonstrated by successful analyses of any combination of at least 3 each of at least two types of spiked QC samples (blank spikes, matrix spikes or lab control samples). These may be documented on the forms in Appendix B and the training forms in Appendix C but a summary must accompany the certifying form which either includes or provides reference to the data.

4.1.6 It is acceptable to certify such capabilities on multiple forms and to certify for multiple analysis types and/or methods on one form. At the time of this writing, there may be no known materials which can be submitted as unknowns for some analyses. In this event, at the discretion of the Division Manager and Quality Assurance Officer, this form may also be used to qualify analysts. From the date of the first version of this SOP, however, this should not be done where materials are readily available and reasonably handled.

4.1.7 When this process is completed, the original of this form and a copy of all applicable documentation will be inserted into the analyst's training file which is maintained in the QA area for the 940 building and the Extractions Supervisor Office for the 921 building.

4.2 Demonstration of Capability to Perform Analysis

4.2.1 For analysts who are relatively new to their assigned tasks, a greater degree of capability demonstration must be undertaken through the satisfactory completion of any internal departmental training documentation. This training will include specific training and documentation developed by that department and department manager and may include required reading, quizzes, and performance criteria at the discretion of the department manager and QA. Example checklists are provided as Appendix C.

4.2.2 In general, if an analyst has not passed the criteria detailed in 4.1, then he/she must proceed through the following:

4.2.2.1 A trainer is designated for the task/test

4.2.2.2 One-on-one training occurs for the timeframe designated by the supervisor and applicable checklists.

4.2.2.3 Training may also include required reading of SOPs and the QA Plan, quizzes, and subset task demonstrations.

4.2.2.4 Progress is monitored and documented on applicable forms.

4.2.2.5 Supervised training continues until the analyst is deemed ready for capability demonstration.

4.2.2.6 Demonstration of analytical competency completion, however, will be the same. Performance Evaluation and/or P&A elements as described previously in 4.1.3, 4.1.4 *or 4.1.5.*

4.2.3 Where P&A demonstration is not required and defined by the method, Laucks may choose to apply additional internal P&A criteria similar to a typical P&A study. The samples may be submitted by the QC Officer, the Division Manager, or an individual designated by one of the above. Four or more aliquots of a material will be submitted to the analyst as unknowns. The analyst must demonstrate the capability to achieve results within the recovery range specified by the manufacturer, if they are independent materials, or within laboratory recovery criteria if they are prepared in-house. In addition, the % RSD of the results must be within Laucks established RPD limits (or default RPDs if none exist for a specific target analyte).

4.2.4 It is recognized that some independent materials may not recover within manufacturers criteria, at least for a subset of the target analyte list, regardless of the experience and competence of the analyst, due to degradation of the material, arbitrary setting of the limits, determination of the "true" values by methods other than those used for the analysis, or other factors. In that case, the % RSD may be the major factor in evaluation and other considerations or action may be taken at the discretion of the QC Officer and/or Division Manager, such as how Laucks more experienced analysts have historically performed for a particular material.

4.2.5 Failure to meet criteria means that the analyst must continue to work under the close supervision of a trained analyst.

4.2.6 Likewise, meeting these criteria may be determined to be only one step in the overall training process. Whereas this is demonstration that the analyst is capable of obtaining reliable results, the Division Manager or other supervisory personnel may determine that a more complete knowledge of the analytical process is in order, such as instrument maintenance capabilities, method troubleshooting, data reduction, proven performance on actual sample analysis, etc.

4.2.7 When such materials are analyzed, a <u>Demonstration of Capability to Perform Analysis</u> form is completed (see Appendix B). This form is designed for single analyte methods. For multi-analyte materials, a page may be attached which depicts all of the analyst's results and the control criteria. However, this is the final signature form and must accompany any summary pages or written evaluation which may be considered pertinent. Also attached should be copies of the supporting data or a data summary page which references the workorder under which the data may be found.

4.2.8 The date of analysis, the results, the recoveries, and the % RSD are recorded on the form (or the attached summary). If all analytes met or did not meet criteria, the appropriate box is checked. If not all criteria are met but the analyst was considered to have performed adequately, a narrative explanation must accompany the evaluation, either on the back of the form or as a separate, attached report.

4.2.9 Additionally, if the analyst, through the analysis of these samples is considered fully qualified to perform the analysis, the appropriate box is checked and the form signed by the Division Manager. If the Division Manager considers that the analyst is now capable of analysis but still requires additional experience and training before they are fully capable of independent analysis, a date is set to review performance. The additional experience or training required and the next performance review date are recorded on the form (with the appropriate box checked) and initialed.

4.2.10 Alternatively, training checklists for many tasks have been developed. In the case of analytical duties, these may include reference to the completion of P&A or PE studies or individual QC analyses as previously discussed. Completion of one of these training checklists (including appropriate signatures) is considered demonstration of training. However, PE or P&A results or other appropriate documentation should also accompany the training checklist as demonstration of competency.

4.2.11 If further training is still required, copies of these forms will be retained by QA in a file to be reviewed regularly to insure that this final analyst review occurs in a timely fashion. A copy of the form indicating interim status will also be retained in the staff member's training file.

4.2.12 When this process is completed, the original of this form will be inserted into the analyst's training files.

4.3 Ongoing Demonstration of Performance

4.3.1 At least annually, after initial qualification, analyst proficiency must be demonstrated. Each staff member that performs a method must demonstrate their continued proficiency through analysis of single blind proficiency samples (another PE). WP, WS or commercial PE samples may be used to satisfy this requirement just as they were used for initial qualification.

4.3.2 As with initial qualification, continuing performance must be documented in the analyst's training file. Ongoing competency can be documented using the Recognition of Experience and Training Form.

5. References

Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center, February 1996

Laucks SOP
LTL-1011 Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits

Appendix A

Recognition of Experience and Training Form

Recognition of Experience & Training Form

Laucks Testing Laboratories

It is hereby recognized that ____

Employee Name

has demonstrated competence in the methodologies listed below. Through the successful analysis of numerous samples, including performance evaluation samples, matrix spikes, laboratory control samples, etc. and in the associated reduction of data as required by these methods, we certify this staff member as being capable of independent performance of the listed analyses.

Analysis Type	Method Numbers	Has Been Performing Analyses by These Methods Since	Has Demonstrated Competency by meeting the following criteria, with the hard copy of applicable information relating to this competency attached to this form

______________________________________ Division Manager

______________________ Date

Appendix B

Demonstration of Capability to Perform Analysis Form

Demonstration of Capability to Perform Analysis

Laucks Testing Laboratories

Analyst: ______________________________

The above analyst has independently analyzed at least 4 aliquots of the listed performance evaluation material, which were submitted as blind samples, achieving the listed recoveries. The limits specified by the manufacturer are considered within acceptable range or, if prepared by Laucks from known materials, the laboratory established control limits apply. In addition, the % relative standard deviation (%RSD) of these data is evaluated against the laboratory established RPD limits as set at the time of this evaluation.

Method: ______________ PE Material: ______________________________

Target Value: ______________ Recovery Criteria: ______________________

Reproducibility Criterion: ______________________

Date	Result	% Recovery

Criteria for non-analytical functions: ______________________________
Demonstrated by: ______________________________

Met Criteria Did Not Meet Criteria

These data are considered adequate demonstration of independent performance if all criteria are met. Other factors may prevail, at the discretion of the appropriate Division Manager before any analyst may be allowed to independently analyze actual samples.

Analyst has met performance criteria but requires more experience. Specific areas which require further training or experience are ______________________________
Work will be reviewed in ______________ and capabilities evaluated. *[Initial here. Do not sign below]*

Analyst has met performance criteria and has been found fully capable of independent work. *[Sign Below]*

______________________________ ______________

Division Manager Date

Appendix C

Example Training Checklists

Laucks Testing Labs

Pesticide/Herbicide GC Semivolatile Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s): ______________________________				
5	Has read and understands EPA Methods (SW846, CLP, 500 & 600 series)				
	List Method(s): ______________________________				
6	Has read and understands appropriate sections of GC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic GC theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to change syringe, septa & injection port liner				
12	Able to trim/change columns, install Y connector & perform leak check				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	NON-ROUTINE: *Able to change detectors*				
16	NON-ROUTINE: *Able to perform total system cleaning*				
Analytical Performance					
17	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze breakdown check and apply QC acceptance criteria				
19	Able to analyze and generate acceptable calibration curve				
20	Able to analyze CCVs and apply QC acceptance criteria				
21	Applies acceptance criteria for surrogates and spikes				
22	Able to set up analytical runs (CLP & non-CLP) & acquire data				
23	Able to get information on samples/analyses (test codes, MDLs, etc.)				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows how to confirm detection of analytes (peak ID, conf. col.)				
26	Knows reanalysis and reextraction criteria				
27	Able to perform sample dilutions (obtaining linear results)				
28	Knows correct reporting limits for method(s)				
29	Knows corrective action & documentation for out of control QC events				
30	Able to produce a data package (In-house, CLP and SW-846)				
Method Validation *(complete one or more of the following)*					
31	Has successfully analyzed four P&A samples				
32	Has successfully analyzed two PE samples				
33	Has successfully analyzed three each of two types of QC samples				

This is to certify that ______________________________ has been an analyst in the GC semivolatile department and has demonstrated competency at the preceeding tasks for the following methods (list below):

____

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

GC Volatile Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s): ______________________				
5	Has read and understands EPA Methods				
	List Method(s): ______________________				
6	Has read and understands appropriate sections of GC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic GC theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to check system flows				
12	Able to trim/change columns & perform leak check				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	NON-ROUTINE: *Able to clean P&T and autosampler lines*				
16	NON-ROUTINE: *Able to change nickel tubing, resin and IPA*				
Analytical Performance					
17	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze and generate acceptable calibration curve				
19	Able to analyze CCVs and apply QC acceptance criteria				
20	Applies acceptance criteria for surrogates and spikes				
21	Able to set up analytical runs & acquire data				
22	Able to get information on samples/analyses (test codes, MDLs, etc.)				
23	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
24	Knows how to confirm detection of analytes (peak ID, conf. col.)				
25	Knows reanalysis criteria				
26	Able to perform sample dilutions (obtaining linear results)				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to produce a data package (In-house and SW-846)				
Method Validation *(complete one or more of the following)*					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

This is to certify that ______________________ has been an analyst in the GC volatile department and has demonstrated competency at the preceeding tasks for the following methods (list below):

____

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

Laucks Testing Labs
Fuels GC Semivolatile Analyst Training Verification Checklist

	Analyst Name:	Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s): ______________________				
5	Has read and understands EPA & State Methods				
	List Method(s): ______________________				
6	Has read and understands appropriate sections of GC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic GC theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to change syringe, septa & injection port liner				
12	Able to trim/change columns & perform leak check				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	**NON-ROUTINE:** *Able to change detectors*				
16	**NON-ROUTINE:** *Able to perform total system cleaning*				
Analytical Performance					
17	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze RTM standard and set up elution range				
19	Able to analyze and generate acceptable calibration curve				
20	Able to analyze CCVs and apply QC acceptance criteria				
21	Applies acceptance criteria for surrogates and spikes				
22	Able to set up analytical runs & acquire data				
23	Able to get information on samples/analyses (test codes, MDLs, etc.)				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows reanalysis and reextraction criteria				
26	Able to perform sample dilutions (obtaining linear results)				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to produce a data package (In-house and SW-846)				
Method Validation *(complete one or more of the following)*					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

This is to certify that ______________________ has been an analyst in the GC semivolatile department and has demonstrated competency at the preceeding tasks for the following methods (list below):

____

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

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HPLC Semivolatile Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	*List Method(s):*________________________				
5	Has read and understands EPA Methods (SW846)				
	*List Method(s):*________________________				
6	Has read and understands appropriate sections of HPLC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic HPLC theory				
9	Able to use solvent delivery system to set mobile phase program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to change filters and guard column				
12	Able to change columns & perform leak checks				
13	Able to measure and set mobile phase flows				
14	Able to prime pumps				
15	Able to prepare mobile phase (filter water, select correct solvent grade)				
16	Able to change Helium tank				
17	NON-ROUTINE: *Able to change lamps*				
18	NON-ROUTINE: *Able to locate the high pressure build-up*				
19	NON-ROUTINE: *Able to change pump seal*				
20	NON-ROUTINE: *Able to clean flow cell*				
Analytical Performance					
21	Able to prepare standards & pass standard QC acceptance criteria				
22	Able to analyze and generate acceptable calibration curve				
23	Able to analyze CCVs and apply QC acceptance criteria				
24	Applies acceptance criteria for surrogates and spikes				
25	Able to set up analytical runs & acquire data				
26	Able to get information on samples/analyses (test codes, MDLs, etc.)				
27	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
28	Knows how to confirm detection of analytes (peak ID, conf. col.)				
29	Knows reanalysis and reextraction criteria				
30	Able to perform sample dilutions (obtaining linear results)				
31	Knows correct reporting limits for method(s)				
32	Knows corrective action & documentation for out of control QC events				
33	Able to produce a data package (In-house and SW-846)				
Method Validation *(complete one or more of the following)*					
34	Has successfully analyzed four P&A samples				
35	Has successfully analyzed two PE samples				
36	Has successfully analyzed three each of two types of QC samples				

This is to certify that ____________________________ has been an analyst in the HPLC semivolatile department and has demonstrated competency at the preceeding tasks for the following methods (list below):

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

Laucks Testing Labs
GC/MS Volatile Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s): ______________________________				
5	Has read and understands EPA Methods (SW846, CLP, 500 & 600 series)				
	List Method(s): ______________________________				
6	Has read and understands appropriate sections of GC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic GC/MS theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set analysis program				
11	Able to change syringe, septa & injection port liner				
12	Able to trim/change columns & perform leak checks				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	**NON-ROUTINE:** *Able to clean total system including P&T and autosampler*				
16	**NON-ROUTINE:** *Able to clean source*				
Analytical Performance					
17	Able to prepare daily standards				
18	Able to inject BFB				
19	Able to tune instrument to meet method specifications				
20	Able to analyze and generate acceptable calibration curve				
21	Able to analyze CCVs and apply QC acceptance criteria				
22	Applies acceptance criteria for surrogates and spikes				
23	Able to set up analytical runs (CLP & non-CLP) & acquire data				
24	Able to get information on samples/analyses (test codes, MDLs, etc.)				
25	Able to quantitate an analytical batch(standards, CCVs, QC & samples)				
26	Knows how to confirm detection of analytes (rt, spectra confirmation)				
27	Knows reanalysis criteria				
28	Able to perform sample dilutions (obtaining linear results)				
29	Knows correct reporting limits for method(s)				
30	Knows corrective action & documentation for out of control QC events				
31	Able to produce a data package (In-house, CLP and SW-846)				
Method Validation *(complete one or more of the following)*					
32	Has successfully analyzed four P&A samples				
33	Has successfully analyzed two PE samples				
34	Has successfully analyzed three each of two types of QC samples				

This is to certify that ______________________________ has been an analyst in the GC/MS Volatile department and has demonstrated competency at the preceeding tasks for the following methods (list below):

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

Metals ICP/MS Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s): ______				
5	Has read and understands EPA & State Methods				
	List Method(s): ______				
6	Has read and understands appropriate sections ICP/MS Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic ICP/MS theory				
9	Able to use the software to create a method				
10	Able to use the software to set up the autosampler table				
11	Able to perform tuning to meet method criteria				
12	Able to change the vacuum pump oil				
13	Able to change the sample introduction pump tubing				
14	NON-ROUTINE: *Able to clean and/or change the cone*				
15	NON-ROUTINE: *Able to clean and/or change the torch*				
16	NON-ROUTINE: *Able to clean and/or change the filters, and know locations*				
17	NON-ROUTINE: *Able to clean and/or change the copper seals*				
Analytical Performance					
18	Able to prepare standards & pass standard QC acceptance criteria				
19	Able to analyze and generate acceptable calibration curve				
20	Able to analyze CCVs and apply QC acceptance criteria				
21	Applies acceptance criteria for internal standards and spikes				
22	Able to set up analytical runs & acquire data				
23	Able to copy the analytical run file from the hard drive to the network				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows reanalysis and redigestion criteria				
26	Able to perform sample dilutions (obtaining linear results)				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to fix the data file in preparation for producing data package				
Method Validation *(complete one or more of the following)*					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

This is to certify that ______________________ has been an analyst in the Metals ICP/MS department and has demonstrated competency at the preceeding tasks for the following methods (list below):

____

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

Laucks Testing Labs
Mercury Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to enter standards in the logbook				
2	Able to generate an instrument run log				
3	Able to make instrument maintenance logbook entries				
Methods					
4	Has read the SOPs for all applicable methods				
	List SOP(s): LTL-7501				
5	Has read and understands EPA & State Methods				
	List Method(s): 254.2, 7470,7471				
6	Has read the appropriate sections of the FIMS 400 Instruction Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic theory				
9	Able to use the software to create a method				
10	Able to use the software to set up the autosampler table				
11	Able to change the peristaltic pump tubing				
12	Able to clean the spectrophotometer windows				
Analytical Performance					
18	Able to prepare standards & pass standard QC acceptance criteria				
19	Able to analyze and generate an acceptable calibration curve				
20	Able to analyze CCVs and apply QC acceptance criteria				
22	Able to set up an analytical run & acquire data				
23	Able to copy the analytical run file from the hard drive to the network				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows reanalysis and redigestion criteria				
26	Able to perform sample dilutions				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to correct the data file in preparation for producing a data package				
Method Validation *(complete one or more of the following)*					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

This is to certify that ______________________________ has been analyzing samples by the methods stated above and has demonstrated competency at the preceeding tasks for the following methods (list below):

____

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

Sample Custodian Training Verification Checklist

Employee Name:		Date:	Trainer:	Supervisor:	Trainee:
Documentation					
1	Able to use Chain of Custodies for received samples				
2	Able to use Sample Receipt, Discrepancy and Preservation Forms				
3	Able to use Internal Chain of Custody Logs				
4	Able to use Chain of Custodies for subcontracted sample analysis				
Methods					
5	Has read and understands SOPs for all applicable job responsibilities				
	List SOP(s): ______________________				
Sample Receipt Process					
6	Knows how to receive samples				
7	Knows SAM basics, including the use of function keys				
8	Able to create client information				
9	Able to create workorders				
10	Able to enter samples in fraction screen				
11	Able to use global jobs and client jobs				
12	Able to create and use SDGs				
13	Able to complete bottle log in SAM				
14	Knows how and where to store samples once received				
15	NON-ROUTINE: *Able to create global jobs and client jobs*				
Additional Responsibilities					
16	Able to prepare, package and ship samples to subcontract lab				
17	Able to preserve and stock bottles				
18	Able to order bottles				
19	Able to complete bottle orders				
20	Able to ship bottle orders by UPS				
21	Able to ship bottle orders by US Mail				
22	Able to ship bottle orders by Fed Ex				
23	Able to ship bottle orders by local courier				
24	Knows how to dispose of soils and document disposal				
25	Knows how to neutralize and dispose of waters and document disposal				
26	Knows and uses appropriate PPE for handling samples				
27	Able to perform sample splitting				
28	Able to perform sample preservation for volatiles methods				
Validation *(complete all of the following)*					
29	Has successfully logged in samples for one month				
30	Has successfully prepared and shipped bottles by all carriers				
31	Has successfully created client and/or global jobs				
32	Has successfully disposed of samples and documented disposal				

This is to certify that ______________________ has been a sample custodian in the sample receiving department and has demonstrated competency at the preceeding tasks.

Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1005

Title: **Analytical Balances**

Revision history:

Number	Date
LTL-0005 R1	09/25/86
LTL-0005 R2	11/3/87
3	06/22/96
4	5/15/98
5	12/15/99

Revised by: Harry Romberg
Harry Romberg, QA Officer
Date: 12-15-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 12-15-99

Contr... ...cument
No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope

1.1 General

1.1.1 The most important piece of equipment in any analytical laboratory is the analytical balance. The degree of accuracy of the data is directly dependent on the accuracy of weight-prepared standards and samples. The balance should be one of the most cared for instruments in the lab. However this is not often the case.

1.1.2 The purpose of this SOP is to insure the proper use and calibration of all analytical balances in the laboratory. It involves the daily use of a standard weight check and a weekly calibration with a class "S". The results of these checks are logged in a balance logbook, thereby maintaining a record of the accuracy of that balance.

1.1.3 On an annual basis, analytical balances are cleaned and general maintenance performed by a qualified service technician. This process occurs automatically in conjunction with the service provider and Laucks purchasing and QA. It is the intent of this SOP to delineate internal calibration practices and not to provide additional specifics on externally provided service.

2. Equipment List

- Analytical Balance
- Manufacturer's Manual
- Balance Record Book
- Class "S" Weights

3. Safety Precautions

3.1 Safety

3.1.1 So as not to expose themselves or other analysts to potential harm and in order not to cross-contaminate samples, **it is critical that the individual analyst clean the balance and the balance area after each and every use of the balance**.

3.1.2 The analyst must not assume that the person using the balance before them cleaned up after themselves adequately and should check the area thoroughly before using the balance and clean up the area if necessary to maintain safety and reduce potential contamination.

3.1.3 Weighing chemicals and samples is potentially hazardous. The analyst should take every precaution to avoid contact of any of these things with the skin, eyes, or through inhalation. In addition, the analyst should take precautions to see that nearby analysts or those using the balance afterwards are not inadvertently exposed.

4. Operation Procedure

4.1 Balance Setup

4.1.1 Most of the balances used at Laucks are of the electronic variety, although there are some mechanical balances. Although electronic balances tend to be somewhat more rugged than the mechanical variety, they are still subject to many of the same conditions which make the operation of all balances a critical component of their continued functioning.

4.1.2 The analytical balance is a fragile and delicate instrument, the operation of which is subject to shock, temperature and humidity changes. Mishandling and other insults also account for great loss in precision and accuracy (P & A). The following precautions should be observed in order to maintain and prolong the life of the balance.

4.1.3 Analytical balances should be mounted on a heavy, shockproof table, preferably one with a sufficiently large work surface. Although shock is less of a concern with electronic balances, they should still be treated with care. For virtually all of the balances currently used by Laucks, except for some of the less sensitive variety which have no leveling bubble, the balance level should be checked frequently and adjusted as necessary.

4.1.4 Balances should be located away from lab traffic and doors or windows where they might be subjected to drafts, sharp temperature changes and physical shock.

4.1.5 For mechanical balances, when the balance is not in use, the beam should be raised from the knife edges and in the lock (rest) position.

4.1.6 For all balances, nothing should be stored on the pan when the balance is not in use.

4.1.7 All doors to the weighing compartment should be closed.

4.1.8 Special precautions should be taken to avoid spillage of corrosive chemicals on the pan or inside the balance case. The interior should be kept scrupulously clean.

4.2 Balance and Weight Calibration

4.2.1 There are three levels of calibration; daily, weekly, and annual.

4.2.1.1 **Daily** - The daily calibration is done by the <u>**first**</u> <u>**user**</u> of the day. The user places a tare weight on the balance equivalent to a tare typically used on that balance, weighs the daily standard (a class "S" weight typical of the weight used on that balance) and records the weight in the balance record book. If the weight is outside the limits set for the standard, it must be brought to the attention of the area supervisor and QA.

4.2.1.2 **Weekly** - The balance will be checked with a range of class S weights each week by the laboratory balance custodian. If a reading for a given weight exceeds the limits for that weight, the balance custodian will bring it to the attention of the area supervisor and QA.

4.2.1.3 **Annual** - Each balance will receive annual servicing and calibration by a qualified balance service representative.

4.2.2 The weights to be used for checking the balances are Class "S" weights or equivalent. The tare weight is not critical, except that it be accurately recorded.

4.2.2.1 The Class "S" Weights - These are the primary standards for checking the accuracy of the balance. They must be handled with care as they are calibrated and damage to the weights may result in inaccurate balance calibration. These weights must only be touched with the forceps supplied with the weights or with the clean white gloves also kept with the weights.

4.2.2.2 The class "S" weights are sent annually to a qualified weight re-certification service, currently Denver Instruments, although another qualified service is allowable. During this time the calibrations will be suspended or other Class "S" weights used (if available) until the calibrated weights return. In the case of our Seattle lab, two sets of certified weights are in use (one normally residing at the 940 building and one at the 921 building). Normally, these weights are used for calibrating balances in their respective buildings. During the annual class "S" weight checks, one set is sent to be calibrated and the entire Seattle facility uses the other weight set. When the first set is returned, the second is sent out and the entire Seattle office uses the first set until the second has been returned. This operation is coordinated annually by QA.

4.3 Responsibilities

4.3.1 The user is to ensure the following tasks are accomplished during the time he or she uses the balance:

- The balance is <u>clean</u> before use.
- The balance is <u>level</u> before use.
- The balance is <u>clean</u> and <u>level</u> after use.
- All weight has been removed and the balance lock lever has been returned to the proper position (for mechanical balances).

- In addition, all balances should be reset to zero when not in use.
- **Prior to use, the user should insure that the daily calibration check has been done**. If not, he or she must complete the task
- **After use, the user will insure the balance is clean and returned to the proper storage position.**
- The user will report any malfunction or failure of the daily check to the area supervisor.
- The user will mark and not use any balance which has failed calibration.

4.3.2 The balance custodian is the person assigned to perform the weekly calibration checks. The custodian's duties include:

- Performing the weekly calibration check
- Marking any balance which has failed the weekly check
- Informing the area supervisor of any balance which has failed the weekly calibration check.

4.3.3 The area supervisor will ensure that the following tasks are accomplished:

- Weekly and daily calibration checks are being performed. It is particularly important to ensure that if the individual assigned to perform the weekly checks (the balance custodian) is absent, that someone is trained and assigned to this duty.
- That any maintenance is performed for balances which do not meet specifications. This may include contacting others, such as QA, to actually correct the problem.
- That any malfunctioning balance or balance which has failed calibration not be used until it is functioning properly.

4.4 Daily Calibration Check

4.4.1 The first user to use the balance each day is to perform the daily calibration check.

4.4.2 The user will insure he or she is familiar with the operation of the balance according to the manufacturer's manual.

4.4.3 The user will first insure that the balance level is correct by checking the balancing bubble and adjusting the legs of the balance as required.

4.4.4 The user checks the zero of the balance. If it is off, the user will adjust it according to the manufacturer's manual.

4.4.5 The user will place a tare weight on the balance which is typical of weights used on that balance (such as an empty beaker or an empty VOA vial). The weight of the tare should be recorded, strictly for the record, and the balance zeroed on that weight, if it is a balance capable of zeroing on the tare (all electronic balances are so equipped). The weight of the tare is not a controlled value but is only used to indicate the level of the tare used.

4.4.6 A standard weight of a size commonly used on that balance must then be added and the weight relative to the tare recorded under the appropriate day of the week in the calibration logbook. He or she will also initial and date the entry (See Appendix I). The standard weight will be a class "S" weight or equivalent.

4.4.7 The daily weight, after taring, must not vary from its true value by more than the following amounts:

Balance capable of weighing to:	must not vary by more than:
0.1 gram	±0.2 gram
0.01 gram	±0.02 gram
0.001 gram	±0.002 gram
0.0001 gram	±0.0005 gram

4.4.7.1 **Example 1:** 1 gram samples are typically weighed into flasks with tare weights of 100 grams on a balance weighing to 0.0001 g. In order to perform the daily calibration check, a flask of about 100 grams is placed on the balance and the weight recorded. The balance is tared (set to zero) based on this weight. A 1.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This second weight must read within the limits of 0.9995 gm to 1.0005 gm.

4.4.7.2 **Example 2:** 30 gram samples are typically weighed into beakers with tare weights of 80 grams on a balance capable of weighing to 0.01 grams. In order to perform the daily calibration check, a beaker weighing about 80 grams is placed on the balance and the weight recorded. The balance is tared (set to zero) based on this weight. A 30.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This second weight must read within the limits of 29.98 gm to 30.02 gm.

4.4.8 If the user cannot obtain a weight within the control limits established for the standard weight, he or she will bring it to the attention of the area supervisor and QA. Nothing requiring accurate weight should be weighed on a balance that does not meet calibration specifications. Any balance exceeding criteria must be clearly marked until it can be brought into control.

4.4.9 An example logbook page is presented in Appendix I

4.5 Weekly Calibration Check

4.5.1 The balance custodian is the person responsible for performing the weekly calibration check and reporting problems to the area supervisor or QA. The custodian may be a different person in each area and it is the responsibility of the area supervisor to ensure that a capable balance custodian has been assigned to each area for which they are responsible. It is the responsibility of the custodian to insure that the weekly check is done even if they are not present, such as for vacation, etc.

4.5.2 On the first day of the week, the balance custodian will perform a calibration check on each balance in the lab to which they are assigned. The results of these checks will be recorded in each balance calibration logbook. This check will be performed using the laboratory Class "S" weights.

4.5.3 The balance custodian will locate the Class "S" weights and insure they are clean. They will be returned to their proper location immediately upon completion of the calibration checks.

4.5.4 The balance custodian will insure the balance is clean.

4.5.5 The balance custodian checks the zero on the balance. If it is off, he or she will adjust it according to the manufacturer's manual.

4.5.6 At a **minimum**, the balance custodian will weigh 3 weights over the range for which the balance is used. Additional weights should be used if the range used is large in order to span the range typically used for that balance. If a specific weight (i.e. 100 mg or 30 grams) is the most often used on that balance, that weight should be included in the range of calibration. The results will be recorded to the left of the entries for the daily calibration check on separate lines. The custodian will also sign and **date** the entry. The date must include the month, day and year (See Appendix I).

4.5.7 Criteria for the weights on the weekly calibration check are as follows:

	True value of weight			
↓Balance capable of weighing: ↓	<0.1000 - 1.0000	1.0000-9.99	10. - 50.	>50.
0.1 gram	inappropriate	±0.1	±0.2	±0.2
0.01 gram	±0.02	±0.02	±0.02	±0.02
0.001 gram	±0.002	±0.002	±0.002	±0.005
0.0001 gram	±0.0005	±0.0005	±0.0020	±0.0050

4.5.8 If the balance custodian cannot obtain a reading within the control limits established for the standard weights, he or she will bring it to the attention of the area supervisor and QA.

4.5.9 An example logbook page is presented in Appendix I

4.6 Annual Calibration Check

4.6.1 The laboratory employs a reputable outside firm to perform annual maintenance and calibration of all of the analytical balances. The current firm is North West Instrument Services but any reputable vendor may be used if first approved by QA.

5. References

ASTM Standard Method of Testing, TOP-LOADING, DIRECT READING LABORATORY SCALES AND BALANCES, Designation: E 898 - 82

APPENDIX I

Sample Page from a Balance Logbook

	TV	Class "S" Read		Mon.	Tues.	Wed.	Thurs.	Fri	S
4- May 9, 1998	2.0	2.00	Tare	54.17	54.16	54.17	54.18	54.15	
	10.0	10.00	2.0 g.	2.00	2.00	2.00	2.00	2.00	
DHR	50.0	50.00		DHR	DHR	DHR	DHR	DHR	
5-4-98	150.0	150.00		5-4-98	5-5-98	5-6-98	5-7-98	5-8-98	
11- May 16, 1998	2.0	2.00	Tare	54.17					
	10.0	10.00	2.0 g	2.00					
DHR	50.0	50.00		DHR					
5-11-98	150.0	150.00		5-11-98					

Done once per week

Initial & Date required

Row Heading

Daily Weights

Initial & Date required

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1006

Title: Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance

Revision history:

Number	Date
1	05/01/96
2	3/20/98
3	12/21/99

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 12-21-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 12-22-99

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No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP provides a description of the identification and annual calibration of thermometers used for refrigerators, freezers, and ovens and the system used to record the calibrations and locations of the thermometers.

1.1.2 This SOP also provides a description of the routine monitoring, maintenance, and corrective actions to be performed when cold storage units or ovens fail to meet control limits.

2. Equipment List

2.1 Equipment

- NIST Traceable Standard Thermometer with a range of at least -20°C to at least 110°C.
- High temperature grease pen
- Erlenmeyer flask
- ethylene glycol or equivalent solution
- thermometers covering temperatures within the operating range of the cold storage unit, oven, or other equipment of interest.
- Water

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 During the calibration and data recording the analyst will be exposed to minimal safety hazards: boiling water, hot ovens, and mercury filled thermometers. It is incumbent on the analyst to exercise due care and caution while executing this SOP. The company will provide any protective equipment or clothing needed to assure employee safety.

3.2 Waste Disposal

3.2.1 No waste is generated in this operation. If mercury-filled thermometers are broken, however, the mercury must be collected and stored with other elemental mercury so that it may either be used in other laboratory operations or disposed.

4. Thermometer Purchasing and Identification

4.1 Purchasing

4.1.1 Laucks currently has several Streck Laboratories, ERTCO and VWR Brand thermometers for cold storage monitoring but thermometers may be purchased from any reputable supplier.

4.1.2 Thermometers used for sample or standard cold storage should be accompanied by either an actual certificate of calibration against a NIST traceable thermometer or a certificate verifying that a the thermometer was calibrated in accordance with standards traceable to the National Institute of Standards and Technology and does not vary by more than one scale division. They are immersed in a vial of ethylene glycol or equivalent solution to prevent freezing and to stabilize the temperature.

4.1.3 Thermometers used for oven temperature monitoring or for other purposes need to cover the expected range of the unit or process to be measured.

4.2 Identification

4.2.1 Thermometers are received with an individual serial number imprinted on the thermometer or may be identified in any way that distinctly distinguishes them from any other thermometer. This may involve the laboratory marking the thermometer to distinguish it from others if it does not have a distinct serial number. The use of a temperature resistant grease pen may be the most suitable for this purpose but any mechanism may be used as long as the thermometer is distinctly identified.

5. Calibration

5.1 Recalibration of the standard thermometer.

5.1.1 The NIST traceable standard thermometer is recalibrated annually by sending it back to a manufacturer who has the capability to recalibrate thermometers to NIST specifications. Currently, Laucks uses the EverReady Thermometer Company (ERTCO) for recalibration services. This vendor will re-calibrate thermometers at approximately the same points at which the original calibration was performed and will take thermometers from any vendor, as long as a copy of the original calibration certificate is available.

5.1.2 Note: Microbiology NIST traceable thermometers are recalibrated at the frequency required by the Washington State Department of Health, every 3 years.

5.1.3 At a minimum, copies of the certificates of recalibration will be kept in QA files.

5.2 Recalibration of Cold Storage and Room Temperature Thermometers

5.2.1 Refrigerator thermometers are calibrated upon receipt and annually thereafter, shortly after the return of the standard thermometer from its annual recalibration. When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. Thus an observer can easily know his/her thermometer is currently calibrated.

5.2.2 Cold storage thermometers should not be calibrated with the standard thermometer if the standard thermometer has just been used at high temperatures (such as boiling water solutions). Thermal expansion of the thermometer at radically different temperatures may result in inaccuracies. After use at high temperatures, the standard thermometer should be allowed to stabilize at room temperature for at least 24 hours before it is used for cold storage calibration.

5.2.3 Refrigerator thermometers are placed in any functional refrigerator which is not frequently opened and has adequate space and in which the temperature is between +2°C and +6°C. Freezer thermometers are placed in a functional freezer where the temperature is between -10°C and -20°C. The temperature of the refrigerator or freezer is not especially important except that it must be accurately recorded and should be in the approximate range that refrigerators or freezers are generally be kept. Cooler thermometers are already immersed in a small vial of liquid. If a thermometer is not already in such a vial it may be placed in the same Erlenmeyer flask as the standard thermometer noted below.

5.2.4 At the same time the standard thermometer is also placed in the cold storage unit. The standard thermometer is placed in the cooler in an Erlenmeyer flask of water, ethylene glycol or other suitable liquid that will not freeze at the temperature of the unit.

5.2.5 Thermometers used for temperatures near room temperature may be calibrated in the BOD incubator using the same process.

5.2.6 The thermometers are allowed to equilibrate at least overnight (12 hours) and the temperatures read and recorded. Read the temperature of the standard thermometer first, then the individual thermometers.

> **Note:** Most thermometers are marked in 1°C or 2°C increments. This will require interpolation by the analyst to estimate intermediate temperatures.

5.2.7 Temperatures are recorded on a blank hardcopy of an Excel spreadsheet along with the cold storage unit ID and location, the thermometer ID, and the date (See appendix A). The data are later transferred to the electronic version for storage and printing. The standard thermometer and the individual thermometer readings are recorded in the log and the difference is calculated and recorded to the nearest 0.1°C.

5.2.8 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number to the individual thermometer reading will result in a "correct" temperature.

5.2.9 Correction factors are also recorded on the Cold Storage Temperature logs. An example of one of these forms is in Appendix B. The year at the top of this form changes annually without invalidating this SOP. They are located on each cold storage unit which is used for storage of environmental samples or standards.

5.2.10 When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. A similar color coded sticker will be attached to the original hardcopy of the annual calibration log so an observer (with the log) can easily know that the thermometer is currently calibrated. Other thermometers will be marked with tape, the calibration factor noted, and initialed and dated.

5.3 Recalibration of Oven and Other Thermometers

5.3.1 Oven and other warm temperature thermometers should not be calibrated with the standard thermometer if the standard thermometer has just been used at low temperatures (such as refrigerator or freezer calibrations). Thermal expansion of the thermometer at radically different temperatures may result in inaccuracies. After use at low temperatures, the standard thermometer should be allowed to stabilize at room temperature for at least 24 hours before it is used for high temperature calibration.

5.3.2 For hot temperature calibration (generally used at temperatures too hot to touch), thermometers are calibrated in a boiling water bath. The standard and individual thermometers are inserted into a beaker of boiling water up to the immersion line. The thermometers will read a temperature slightly above 100°C if the bulbs of the thermometers are resting directly on the bottom of the beaker while the hotplate is in a heating mode. The thermometers are allowed to equilibrate for four-five minutes and the temperatures read to the nearest 1°C. Temperatures are recorded in an Excel spreadsheet along with the oven ID (if it was an oven thermometer), the thermometer ID, and the date (See appendix A).

5.3.3 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number to the individual thermometer reading will result in a "correct" temperature.

5.3.4 Correction factors may be written on the thermometer or on the unit with which that thermometer is used.

5.3.5 When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. A similar color coded sticker will be attached to the original hardcopy of the annual calibration log so an observer (with the log) can easily know that the thermometer is currently calibrated. Other thermometers will be marked with tape, the calibration factor noted, and initialed and dated.

5.4 Recalibration of the Infrared Thermometer

5.4.1 The standard thermometer is placed in a glass Erlenmeyer flask filled with water in a cold storage unit at least overnight (12 hours).

5.4.2 The infrared thermometer is used to measure the temperature of the flask while it contains the standard thermometer.

5.4.3 The emissivity of the IR thermometer is set at the level determined in the previous calibration (if any). It should read the same temperature as the standard thermometer. If it doesn't, the emissivity is adjusted until the standard thermometer and the IR thermometer agree as closely as possible.

5.4.4 This emissivity setting, the calibration date and the person who performed the calibration are recorded on a label which is attached to the thermometer. Analysts subsequently using the thermometer must measure against a glass container and use the emissivity setting noted on the IR thermometer in order to get an accurate temperature measurement.

5.4.5 The appropriate information is also recorded on the Excel spreadsheet used for the other calibrations.

5.5 Daily Calibration Check of the Infrared Thermometer

5.5.1 Outside of its regular calibration against the NIST traceable thermometer, the infrared thermometer is checked daily at 4°C ± 2°C against a regular glass-column thermometer, which itself has been calibrated as previously discussed.

5.5.2 The regular thermometer is stored in the 940 walk-in (W01) in water in an Erlenmeyer flask. The infrared thermometer is checked against the side of the Erlenmeyer. The infrared thermometer should agree with the regular thermometer (corrected to the NIST thermometer) within ±2°C or the discrepancy further investigated.

5.5.3 Most often, a discrepancy may be corrected by cleaning the surface of the Erlenmeyer and re-taking the measurement or by replacing the IR thermometer's batteries. The latter or any other action must be recorded on the calibration logsheet. If discrepancies persist but are consistent, it may just be necessary to fully recalibrate the infrared thermometer and adjust the approved emissivity setting. This should be coordinated with QA.

6. Monitoring Responsibilities

6.1 Use Of Calibration Logs For Cold Storage Monitoring

6.1.1 At the time of annual calibration of the individual thermometers, the correction factor is written in the space provided on the form by QA. This correction factor (as noted) is calculated such that adding the value results in a temperature corrected to the standard thermometer. This correction factor may be positive or negative depending upon whether the specific individual thermometer read low or high when compared to the standard thermometer.

6.1.2 It is not the intent of this SOP to discuss how individuals whose responsibility it is to monitor cold storage units are assigned. It is the responsibility of departmental supervisors to ensure that this activity is occurring in their areas.

6.1.3 The person monitoring each cold storage unit will add the correction factor to the value read on the thermometer when recording the temperature. The corrected temperature is reported to the nearest 0.1°C. As noted previously, temperatures are estimated between thermometer marks.

6.1.4 The person monitoring each cold storage unit will also check the thermometer to make sure their are no breaks in the column.

6.1.5 It is the responsibility of the person monitoring the particular unit to take corrective action as noted in this SOP and on the monitoring form or to see that corrective action is initiated by informing a supervisor. Any corrective actions (including simple adjustments of the cold storage unit thermostat) **must** be noted on the Cold Storage Temperature Log (Appendix B).

6.1.6 The calibration forms change quarterly, when new cold storage units are put on-line, or when unforeseen circumstances occur which call for a new form. The individual charged with monitoring the cold storage unit will transfer the cold storage ID, the cold storage unit location, the thermometer ID, and the correction factor to the new form. That person will also turn in the completed log to QA for permanent storage.

6.2 Monitoring Ovens And Other Devices

6.2.1 When oven thermometers are calibrated, QA will mark the oven with the thermometer ID and the correction factor. Log sheets are generally not used for ovens. It is the responsibility of any analyst using an oven to apply the correction factor when recording temperatures on data sheets.

6.2.2 In other cases where thermometers are calibrated, the correction factor will be kept with the thermometer or written directly on the thermometer (generally with a piece of tape). Again, it is the responsibility of the analyst to apply the correction factor when recording temperatures on data sheets.

7. Specification Limits and Corrective Actions

7.1 Thermometer Criteria

7.1.1 Thermometers should not vary by more than ±5°C from the standard thermometer reading, even though a correction factor is applied. This criterion does not apply to the infrared thermometer.

7.1.2 There should be no observable breaks in the column of any thermometer at any time during calibration or routine use.

7.2 Thermometer Corrective Actions

7.2.1 Thermometers with a break in the column must be immediately removed from use and either repaired or replaced.

7.2.2 Thermometers which read more than ±5°C from the standard thermometer reading must not be used. If they cannot be repaired or (if new) returned to the vendor, they should be disposed or clearly marked and only used for non-critical tasks. They should not be used for the storage or analysis of environmental samples or others where temperature is a critical factor.

7.3 Cold Storage Criteria

7.3.1 Refrigeration units should be in the temperature range of 4°C ± 2°C. All freezers must be <-10°C.

7.4 Cold Storage Corrective Actions

7.4.1 See Appendix B for an example Cold Storage Temperature Log. This log also contains the appropriate corrective actions in an abbreviated form.

7.4.1.1 If at all possible, actions should be taken __before__ a unit exceeds temperature criteria so that samples or standards are not inadvertently stored outside the required limits for any significant length of time. This alleviates the far more onerous tasks of re-preparing standards or contacting clients for samples stored out-of-specifications.

7.4.2 Adjust the thermostat of the cold storage unit if necessary.

7.4.3 Defrost the cold storage unit if necessary. This may be done prior to adjusting the thermostat if there is severe icing of the unit and it is obvious that this is the cause of the temperature deviation.

7.4.4 If the above fail to correct the problem, contact the laboratory maintenance personnel, the departmental supervisor or QA to arrange for repair.

7.4.5 If it is determined that professional servicing is required this may be arranged upon direction of one of these individuals or another senior supervisor. If professional maintenance does not correct the problem, the unit may need to be replaced, again at management discretion.

7.4.6 Samples must not be stored at inappropriate temperatures. If the problem is not quickly solved, samples or standards must be transferred to another cold storage unit. If it is determined that samples were stored at inappropriate temperatures for an extended period, it may be necessary to contact clients to determine the course of action they would like us to take regarding their analyses. This should be coordinated with QA and project management. Standards which have been inappropriately stored will generally require disposal, generally at the discretion of QA and/or department managers.

7.4.7 Any corrective actions (including simple adjustments) **must** be noted on the Cold Storage Temperature Log (Appendix B).

Appendix I

Example Calibration Spreadsheet

Laucks Testing Laboratories
Thermometer Calibrations
1999

Cold Storage ID	Location	Serial No.	Cal. Date		FC-53631 NIST reading	Thermom. Reading	Correction Factor
C01	Inorganics	ERTCO 4156	05/10/1999		1.9	2.1	-0.2
C02	GC residues	none	05/10/1999		2.0	2.0	0.0
C03	extractions	B B01679	05/10/1999		2.0	1.8	0.2
C04	GC VOA	B B03987	05/10/1999		2.0	2.2	-0.2
C05	Inorganics	B02580	05/10/1999		1.9	1.5	0.4
C06	Warehouse	B B04765	05/10/1999		2.0	1.4	0.6
R02	GC Semi Stds.	B B03534	05/10/1999		2.0	1.8	0.2
R04	GC/MS whse.	B B00906	05/10/1999		2.0	1.9	0.1
R06	extractions	B B01708	05/10/1999		2.0	1.8	0.2
R07	extractions	B B04059	11/15/1999		4.0	4.0	0.0
R08	Inorganics	B03021	05/10/1999		1.9	1.8	0.1
R11	929 warehouse	B B01342	05/10/1999		2.0	2.0	0.0
W01	940 Walk-in	B B03928	05/10/1999		2.0	1.8	0.2
W02	921 Walk-in	B B01919	05/10/1999		2.0	2.1	-0.1
F03	GC semivolatiles	B B B08959	05/11/1999		-11.3	-12.0	0.7
F04	GC semivolatiles	B B B09215	05/11/1999		-11.3	-11.3	0.0
F05	GC/MS VOA	B B B06608	05/11/1999		-11.3	-12.7	1.4
F06	GC/VOA	B B B08543	05/11/1999		-11.3	-12.0	0.7
F07	GC/VOA stds.	B B B08765	05/11/1999		-11.3	-12.0	0.7
		Ertco 5236	05/11/1999		-11.3	-12.4	1.1
F08	929 warehouse	Ertco 4268	05/11/1999		-11.3	-11.3	0.0
		Ertco 5034	05/11/1999		-11.3	-12.5	1.2
Hg thermometer	Sample entry	BCR 2	05/10/1999		2.0	1.8	0.2
IR Thermometer (ITT-330)		Horiba 226099	05/10/1999		1.6	1.7	(at E=82)
spare		VWR 172101	05/10/1999		1.9	0.6	1.3

Oven/Water-bath/Other ID	Location	Serial No.	Cal. Date		FC-53631 NIST reading	Thermom. Reading	Correction Factor
VWR 1320		VWR 61019-204	06/22/1999	retired 6/22/99	101	103	-2.0
VWR 1320	main lab brown hood	F22641	06/22/1999		101	102	-1.0
Thelco		2	06/16/1999		101.0	101.5	-0.5
VWR 1310		3	06/16/1999		101.0	100.0	1.0
VWR 1330F	#4 Univ. Enterprises	L12-004	06/16/1999		101.2	100.8	0.4
VWR 1370F		5	06/16/1999		101.2	101.2	0.0
back waterbath (ASTM 1F)		VWR 02429	06/16/1999		101.2	211.5 F	2.7 F
front waterbath		VWR 61066-046	06/16/1999		101.2	97.2	4.0
VWR 1300 U (TOC rooom)		SPER Sci. 106	06/16/1999		101	102	-1.0

Laucks Testing Laboratories
Thermometer Calibrations
1999

Cold Storage ID	Location	Serial No.	Cal. Date	FC-53631 NIST reading	Thermom. Reading	Correction Factor
Pensky Martin (ASTM 9F)		VWR 61091-001	06/16/1999	100.8	213 F	0.4 F
VWR 1305U	TSS/TDS area	F 14547	06/23/1999	101	100	1.0
VWR 1300 U (Extractions)		F 14506	06/22/1999	100	97	3.0
Blue M (Extractions)		F 14669	06/23/1999	101	102	-1.0
front waterbath		Enviro-Safe 6	11/11/1999	99.5	102	-2.5
BOD thermometer		1	05/21/1999	2.6	2.8	-0.2
Napco Oven		SPER Sci.	06/16/1999	101.2	100.5	0.7
Digestion Area		Polyscience USA	06/16/1999	100.8	103	-2.2
VWP ASTM 9F		09107	06/16/1999	100.8	— 216 F	(-2.6 F)
ERDCO	Setaflash	1SF5531/85A038	06/16/1999	100.8	214 F	(-.6 F)
ERDCO	Setaflash	1SFA5531/151831	06/16/1999	100.8	213.4 F	0.0 F
spare		F22716	06/22/1999	100	100	0.0
spare		F22592	06/22/1999	101	98	3.0
spare		F22731	06/22/1999	101	97	4.0

Appendix II

Example Cold Storage Temperature Log

Cold Storage Temperature Log
Laucks Testing Laboratories, Inc.

Cold Storage ID #:_________ Thermometer ID:_________

Location:________________ Year: **2000** Correction Factor (*add this number when **recording** the thermometer reading*):_______°C

	Month:				Month:				Month:			
Day	**Time**	**Temp.**	**Initials**	**Actions**	**Time**	**Temp.**	**Initials**	**Actions**	**Time**	**Temp.**	**Initials**	**Actions**
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
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16												
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20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation. Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/21/99

If refrigerator temperatures exceed 4°C±2°C or if freezer temperatures are warmer than -10°C, corrective action must be taken.

Corrective action includes
1) Adjust the temperature of the thermostat
2) Defrost the refrigerator or freezer
3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer
4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of.

Any and all actions <u>MUST</u> be recorded on this log sheet. If there is insufficient room, mark on the back of the page with the date the action occurred.

Samples <u>MUST NOT</u> be stored in units which are not maintaining the proper temperature.

Appendix III

Example Infrared Thermometer Daily Check Log

Infrared Thermometer Daily Check Log

Laucks Testing Laboratories, Inc.

Thermometer ID: Horiba 226099 Checked against standard glass thermometer: BCR 2

Location: Sample Receiving Year: **2000** Emissivity Setting: 82

	Month:					Month:				
Day	**Time**	**Glass Thermometer Temp. (°C)**	**IR Thermometer Temp. (°C)**	**Initials**	**Actions**	**Time**	**Glass Thermometer Temp. (°C)**	**IR Thermometer Temp. (°C)**	**Initials**	**Actions**
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
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21										
22										
23										
24										
25										
26										
27										
28										
29										
30										
31										

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.
If IR thermometer exceeds ±2°C of the standard glass thermometer, corrective action must be taken. Corrective action includes
1) Change batteries 2) Clean erlenmeyer surface and read again 3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer
4) If the IR thermometer is not working properly, the glass thermometer <u>must</u> be used.
... the back of the page with the date the action occurred.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1007

Title: Use of Instrument Records and Logbooks

Revision history:

Number	Date
1.0	9/5/91
2	5/13/98

Revised by: Harry Romberg
Harry Romberg, QA Officer

Date: 5-13-98

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director

Date: 5-18-98

Controlled Document

No. 20 **Assigned to:** Tetra

Table of Contents

1. Introduction and Scope

1.1 Introduction

1.1.1 The maintenance of instrument logbooks is essential to monitoring instrument performance and throughput and in tracking analyses. It is also important to confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. Several systems are in place at Laucks, the differences being primarily dependent on the specific instrument and analysis types. This SOP will discuss what is expected in each.

1.2 Scope

1.2.1 This SOP primarily addresses instrument run log maintenance. maintenance manuals and other logs not addressed in other SOPs. Standards log, for instance, are discussed in the standards SOP, LTL-1013. Analytical balance logs are discussed in that SOP. LTL-1005. Cold storage logs are discussed in LTL-1006. Control and monitoring of logbooks and general items pertinent to all logbooks is discussed in Laucks SOP LTL-1019.

1.3 Definition of Terms

1.3.1 Logbook - Any bound or unbound document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs. standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs. or any other regularly maintained record of activity.

2. Equipment List and Standards

2.1 Equipment

2.1.1 maintenance logbook. analytical run logbook (where appropriate) or other applicable logbook

2.1.2 pen (pencil is NOT allowed)

3. Operation procedures

3.1 All Logbooks

3.1.1 All logbook. should be numbered and controlled according to procedures outlined in Laucks SOP LTL-1019. It is the analysts responsibility before initiating any new logbook to ensure that the logbook has been identified and given a logbook number by QA. See LTL-1019 for further detail.

3.1.2	**NOTE:** All errors in all logbooks must be altered by a single-line crossout which must also be initialed and dated. No erasures, overwriting, white-out or multiple-line crossouts (blacking out) are acceptable.

3.1.3	**NOTE:** Empty space in logbooks must be lined out (preferably with a Z for large blocks of empty space). This mark, as with error correction, should be initialed and dated.

3.2 Maintenance Manuals

3.2.1 All instruments at Laucks from GC or GC/MS systems to ICPs, AAs. spectrophotometers, ion chromatographs, etc. have instrument maintenance manuals associated with the specific instrument.

3.2.2 Maintenance manuals are bound notebooks with the specific instrument and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which may be the case for instruments which have been in service for a long time, especially if they have required extensive, ongoing maintenance, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.2.3 As a general rule, loose leaf or 3-ring bound notebooks are not acceptable. The exception to this rule is for maintaining copies of professional service call paperwork or if specific forms have been created for monitoring maintenance activities. Such paperwork must be dated. Note of the service should still be made in the bound notebook associated with that instrument and the identifying number on the service log noted in the maintenance manual.

3.2.4 With a few basic rules, these maintenance manuals are free-form with no specific format but MUST include any and all maintenance associated with the particular instrument.

3.2.4.1 Each entry should be INITIALED by the person making the entry.

3.2.4.2 The maintenance manual must contain the DATE any service or maintenance was performed on the instrument and exactly WHAT that operation was. This includes everything from changing a part to cleaning an instrument orifice or changing a chromatographic column or instrument tubing. It should include everything from the simplest maintenance to the most complex, including any professional service calls.

3.2.4.3 Where maintenance is routine, some books use codes for the most common service operations. These codes must be clearly defined either on the front, inside cover of the maintenance manual or on the first page. If there are multiple books, these codes must be so defined in EACH book.

3.2.5 If the maintenance was performed because of a specific problem (not just routine, ongoing maintenance) the problem should be described in at least one entry in the maintenance book as well as the work performed at any one time, and the outcome of that maintenance, that is whether or not it was successful or what occurred when the work was performed.

3.2.6 In order to aid in monitoring instrument performance changes, service or equipment changes may also be noted in instrument run logs. However, this information is supplementary. ALL maintenance must be recorded in the maintenance manual.

3.3 Instrument Run-Logs

3.3.1 Instrument run-logs come in two essentially different forms, with variations depending upon the specific instrument. In any form, a copy of the daily run log must accompany the data from each laboratory workorder for any samples associated with that sequence.

3.3.2 GC, GC/MS, HPLC, GPC and other run-logs are in bound, pre-printed, sequentially page-numbered books. They are identified by the specific instrument type and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which will be the case for instruments which have been in service for very long, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.3.2.1 Run logs must identify the method being run either at the top of the page, or if more than one method is being used for any sequence, clearly marked by the sample entry. It is recognized that it is in some cases possible to use different methods, which may only be different in the way a calibration is interpreted or validated. It may even be that two methods are essentially identical. However, in these instances, the logsheet should clearly indicate for which method a particular sample is being analyzed.

3.3.2.2 Instrument run-logs should include places to record all relevant sample and data file IDs, performance criteria, sample type and size, additional comments pertinent to the specific analyses, and analyst initials. All appropriate information must be filled out and the page dated. Examples of current logbook forms (at the time of this writing) are located in Appendices I (GC/MS), II (GC and HPLC), and III (GPC). These forms should be considered examples and not as the only forms used by Laucks for this purpose. These forms may change with approval of the department manager and QA. Although this SOP will not then be considered invalid, new example forms should be incorporated into the next revision.

3.3.3 In addition to the appropriate header information for each analytical GC, GC/MS, HPLC, GPC or other run, all of the pertinent information should be filled out for each injection.

3.3.4 The standards, samples, calibration checks, reference materials, QC samples, etc. should be listed <u>in the order that they were analyzed</u>.

3.3.5 Logbook information should be either completely filled out, or a logbook designed to incorporate all of the pertinent elements for that analysis so that all fields are filled in. Logbooks should contain all of the necessary information to track what analyses occurred, the processing order, and critical run parameters (such as what GC column was in use).

3.3.6 No empty space should be left between daily logbook entries. The end of the analytical sequence should be clearly marked and empty space on the page crossed out, the accepted practice being with a "Z" which covers the entire space being crossed out. This "Z" should be initialed and dated by the analyst making it.

3.3.7 The other type of run-log typically in use is the individual, loose-leaf instrument run-log printout. Where the instruments themselves don't produce such printouts, handwritten run-logs are produced by the analyst. These are the log types typically in use in the Inorganics area of the laboratory.

3.3.8 A copy of the run-log is included with each data packet associated with that run.

3.3.9 As with the bound book format, the samples, standards, calibration checks, reference materials, etc. should be identified and listed IN ORDER.

3.3.10 Information critical to identifying the analytical run (date, analyst, analysis type) must be included in the header information. If multiple analytical runs were made in one day, they must be identified as run #1, run #2, etc. If the instrument is capable of time-stamping run data, this option should be utilized, although it need not be included in the run-log itself.

3.3.11 Where possible laboratory practice is to maintain ongoing run-logs for inorganic instrumentation. The daily run-logs are included with all data. Records which do not lend themselves to being kept in a pre-printed bound logbook may be collected in a 3-ring binder in an organized format but <u>not</u> unbound or loose-leaf. After sufficient logs have been collected, they should be bound with the laboratory comb binder. These logs should be given QA logbook IDs as described in Laucks SOP LTL-1019.

3.4 Other Logbooks

3.4.1 The same general principals used for the above logbooks apply to any other logbook, unless otherwise defined in a specific SOP.

3.4.1.1 Entries should be initialed and dated.

3.4.1.2 Empty space between entries should be minimized

3.4.1.3 Errors and empty spaces should be properly crossed out, initialed and dated.

3.4.1.4 Pages are preferably sequentially numbered but if this is not practical, at least dated and/or time stamped.

3.4.1.5 The logbook should identify the operation being monitored.

3.4.1.6 The pages in the logbook should contain all appropriate information needed to identify the activity and all applicable spaces should be completely filled out.

3.4.1.7 The logbook should be given a QA ID number as described in LTL-1019.

Appendix I

GC/MS Run Logs

Laucks T[illegible]g Labs, Inc.
GC/MS VOA [illegible]ument Run Log

IS /SS Amt: 1µl of __________ µg/ml
IS /SS Ref # : _______________
Date : ____________

Analyst: _________
Calibration Std Ref.: ____________
Spike Sol'n Ref.: _______________

624 8260 524 SIM CLP-Low CLP

#	FILE ID	SAMPLE ID	INJ. TIME	SAMPLE AMT	DF[1]	pH[2]	COMMENTS

[1] DF = 1 unless otherwise indicated.
[2] pH = 2 for waters unless otherwise noted.

Laucks Testing Laboratories

GC/MS Semivolatiles Injection Logbook

IS Std ID________________

CCV ID________________

DFTPP ID________________

Page: 5699

Date:________________

	File ID	Lab ID	Inj. Time	Sample Info	Dilution	Comments	Analys
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							

Appendix II

GC/HPLC Run Logs

Laucks Testing Labs

GC VOA Instrument Log

Analyst : ____________________ Date : ________________________

Chromatography Ref. Page : ______________________________ Calibration Reference : ________________________

File #	Sample ID	pH	Sample Vol/Wt	STD ID / AMT	SUR ID	Comments

LAUCKS TESTING LABORATORIES

INSTRUMENT LOGSHEET

Analyst : ____________

Date : ________________

Instrument ID : ____________

Column 1: ____________

Injection Volume : ________ ul

Column 2: ____________

Calibration Standard Reference : ________________

File ID	Sample ID	DF	Comments

LAUCKS TESTING LABORATORIES

HPLC INSTRUMENT LOGSHEET

Analyst : ____________ Date : ________________

Instrument ID : ____________ Column : ______________

Injection Volume : ________ ul Solvent : _____________

Calibration Standard Reference : __________________

File ID	Sample ID	DF	Matrix	Comments

Appendix III

GPC Run Log

Laucks Testing Labs, Inc.
GPC Bench Sheet

Date	
Cal. Ref	
Analyst	
Case #	
SDG #	
Matrix	
Program	
Load Time	
Dump Time	
Collect Time	
Wash Time	

Operating Conditions:

N2 Pressure		
tank, high		psi
tank, low		psi
line		psi
Rinse Press.		psi
Sys. Press.		psi
Flow Rate		ml/min
Temp		deg F
Chart Speed		cm/min
Chart Full Scale		Volts
UV Detector		AUFS
Column ID		

Port	LTL Number	Client ID	Intermed Vol (ml)	Aliquot Clean (ml)	Volume Collect (ml)	Final Vol (ml)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						

Comments:

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

Revision history:

Number	Date
5	6/22/96
4	3/3/96
3 (LTL-0008)	6/29/89
2 (LTL-0008)	5/19/87
1 (LTL-0008)	12/12/86

Written by: Harry Romberg
Harry Romberg, Quality Assurance Officer

Date: 6-25-96

Approved by: Karen J Kotz
Karen Kotz, Laboratory Director

Date: 6/25/96

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to establish a system to identify, document and resolve out-of-control events.

1.2 Scope

1.2.1 An out-of-control event may be recognized by any member of Laucks. When they occur, the analyst, supervisor and Quality Assurance work jointly to solve and correct the problem. Out-of-control events are documented using an Out-of-Control-Event form or a Corrective Action form, or in a few selected instances, on a logsheet with space specifically for such actions. Corrective action resulting from an audit is also dealt with using its own Audit Response form but this action is elucidated in an SOP specific to that process.

2. Definition of Terms

2.1 This section defines terms and acronyms as they are used in this SOP.

2.1.1 **Corrective Action:** Action taken by an individual(s) to correct a problem as evidenced by either the failure of QC criteria or a more general problem which could affect performance of an analysis, the quality of service or other activity undertaken by the laboratory.

2.1.2 **Out-of-control event:** Any occurrence or condition failing to meet Laucks QC criteria or has the potential to impact data quality.

2.1.3 **QA/QC:** Quality Assurance/Quality Control

2.1.4 **Reagent blank:** a measured volume of reagents used in a method.

2.1.5 **Method blank:** a reagent blank that undergoes a preparation (digestion, extraction, distillation, etc.) step prior to analysis.

2.1.6 **RPD:** Relative Percent Difference

2.1.7 **LCS:** Laboratory Control Sample

3. OUT-OF-CONTROL EVENT PROCEDURE

3.1 Identifying an Out-Of-Control Event

3.1.1 The following is a list of examples of out-of-control events. This is not a complete list of all possible out-of-control events and many of those listed may be different for different methods. Specific criteria are given in analytical SOPs or in other QA documents. If there is doubt about whether a situation is out-of-control and must be responded to, consult with Quality Assurance.

3.1.1.1 GC/MS instrument tune criteria failing to meet criteria

3.1.1.2 Initial calibration linearity, depending upon the method used for calibration, correlation coefficient <0.995 (<0.990 for some fuels analyses) or percent RSD failing to meet method specifications.

3.1.1.3 Daily and continuing calibration verification or calibration blanks outside acceptable ranges as defined in their respective SOPs.

3.1.1.4 **NOTE:** If any of the above instances (3.1.1.1-3.1.1.3) occurs, analysis is stopped. No sample analysis can occur until the event is back in control. A corrective action form does not need to be filled out for these instances if identified at the analyst level and corrected before any data are affected.

3.1.1.5 Matrix spike, surrogate spike or blank spike recoveries outside acceptable ranges.

3.1.1.6 Unacceptable RPD value for MS/MSD or duplicate samples.

3.1.1.7 Unacceptable values for LCS's and QC samples.

3.1.1.8 A reagent blank containing a target analyte greater than the method reporting limit.

3.1.1.9 A method blank containing interference or a target analyte at a concentration greater than or equal to the method reporting limit.

3.1.1.10 **Note:** Samples which contain target analyte levels which are greater than 20 times the blank or which contain none of the offending analyte may be considered acceptable.

3.1.1.11 A sample received, prepared or analyzed past holding time.

3.1.1.12 A sample depleted before all required analyses are completed.

3.1.1.13 An extract blown down to dryness, spilled or otherwise compromised.

3.1.1.14 Contaminated reagents and glassware.

3.1.1.15 Equipment malfunction or instrument failure, such as cold storage unit temperature outside acceptable ranges and the loss of data acquisition.

3.1.1.16 Record keeping omissions, errors, and deviations from the record keeping standard operating procedures are also out-of-control situations

3.2 Responding to an Out-Of-Control Event

3.2.1 When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

3.2.2 Analyst:

3.2.2.1 Must be able to recognize QC failure and immediately take the proper action or, if unsure of the appropriate response, notify the supervisor and work with the supervisor and Quality Assurance to solve the problem; also maintains QC charts.

3.2.2.2 The analyst is also responsible for performing the following steps to correct the problem:

3.2.2.3 Examine all calculations for correctness

3.2.2.4 Examine bench sheets for correctness

3.2.2.5 Check instrumentation and operating conditions to preclude the possibility of malfunctions or operator error

3.2.2.6 Verify integrity of spiking solution, laboratory control sample, or calibration standard

3.2.2.7 Re-analyze the sample

3.2.2.8 Take other actions as noted in the specific analytical SOP.

3.2.2.9 If these steps do not yield acceptable results, consult the supervisor.

3.2.3 Supervisor:

3.2.3.1 Must review all analytical and QC data for reasonableness, accuracy and clerical errors; also responsible for QC charts. Some of the above duties may be assigned to others, with supervisory oversight, if those others have been trained to observe the conditions which would initiate further investigation.

3.2.3.2 In an out-of-control event, the supervisor works with the analyst and Quality Assurance to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and insuring that all results that are suspect are repeated, if possible, after the source of the error is determined and remedied.

3.2.3.3 If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions and if it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client.

3.2.4 Quality Assurance:

3.2.4.1 The Quality Assurance Officer or designee will work with supervisory personnel and/or analysts to solve out-of-control situations which are not routinely corrected at the bench.

3.2.4.2 In the event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level, such as performance failure on a blind QC sample, Quality Assurance will notify the supervisor, help identify and solve the problem where applicable, insure the work is stopped on the analysis and no suspect data is reported.

3.2.4.3 Finally the Quality Assurance Officer or designee must review and approve all corrective action reports which cannot be resolved. If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions.

3.2.4.4 If it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client and QA will review said comments.

3.2.5 Project Manager:

3.2.5.1 The Project Manager is responsible for notifying the client of out-of-control events, such as missed holding times, raised reporting limits, matrix interferences, etc. which cannot be resolved without potential impact on either the data quality, the agreed upon or routinely reported results, or the timely and expected delivery date. It is not necessary to contact the client for events which are correctable and do not impact the final data quality, holding times or turn-around unless specifically requested by the client.

3.3 Corrective Actions

3.3.1 Appropriate corrective action depends on the type of analysis, the extent of the discrepancy, and whether the event is determinant or not. The corrective action to be taken for analytical QC failures is usually described in the specific analytical method but may also be determined by either the supervisor, Quality Assurance Officer, or by both in conference, if necessary.

3.3.1.1 Some items may not necessitate direct intervention of QA where standard practices are in place for some events, where the SOP or project or program QAP itself dictates the corrective action and where the action taken is the most conservative response practical. These types of events may be considered to have automatic QA approval and may not even require the completion of any related out-of-control event forms.

3.3.2 A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, re-extracting and re-analyzing a complete batch of samples, due to reagent blank contamination; or as simple as recalculating a series of results because a wrong dilution factor was applied. Again, the appropriate corrective action must be determined on a case by case basis.

3.3.3 Data cannot be released until the system is in control or the QC failure can be attributed to a cause other than method performance. In the event the out-of-control event is due to matrix problems in the sample, and the system remained out of control, the data is flagged and supporting documentation is released to the client.

3.3.4 Corrective actions are considered adequate when the problem has been resolved and data can be reported or other actions taken from an in-control condition. Alternatively, it may be determined that the action taken was, as a minimum, all that was required by the method or that no further action was reasonable or possible that would improve the data. In these cases, the final decision must be approved by the supervisor and QA.

3.4 Documenting an Out-Of-Control Event

3.4.1 This is accomplished by completing one of the following

- A Corrective Action (CA) Form (See Appendix 1)
- A QC_DB Report Form (for Inorganics analytical QC only, see Appendix 2)
- An Out-Of-Control Event (OOCE) Form (lab use only, see Appendix 3)
- A Sample Receipt Form (for sample receipt events, see Appendix 4)
- An Audit Finding Report Form (QA use only, not shown here, see audit SOP)
- or logged onto a form which itself includes corrective actions (example, Cold Storage Logsheet, see Appendix 5).

3.4.2 CA forms are general and are for documenting corrective action taken to correct problems not associated with a particular analytical event.

3.4.3 Out-Of-Control Event (OOCE) Forms are filled out by <u>technical</u> laboratory staff only and are designed for documenting analytical QC failures and associated corrective actions. Where other forms, such as the Inorganics QC_DB Report Form, are used to document that the QC parameters were checked, any failures of QC and the decision to perform corrective action or continue data processing must be documented on the OOCE form. The checklist may then be attached to the OOCE form for final data submission.

Note: It is not necessary for analytical staff to document actions which were taken prior to processing samples or which do not affect reported data.

3.4.4 Audit Finding Reports are responded to by the assigned individual and signed off by QA or a designated individual (see the audit SOP).

3.4.5 All OOCE and Corrective Action Forms shall be filled in completely by the person observing the event. Actions taken may be filled in by either the initiating person or the person actually performing the corrective action. The descriptions of the event and any corrective actions taken should be detailed and specific. The OOCE form provides check boxes for most analytical events.

Note: Holding time violations due to laboratory error are annotated on the OOCE form. Holding time violations occurring due to receipt of samples beyond the criteria are documented on the sample receipt form only.

3.4.6 If the corrective action taken and annotated on the OOCE Form resolves the problem and allows data to be reported which is in control, the action is complete and only needs to be signed by the individual taking action and the individual initiating the action.

3.4.7 If the corrective action taken and annotated on the OOCE Form does not resolve the event and it is determined that no further action can or will be taken, the form must be signed by the analyst, supervisor, and QA.

3.4.8 Originals of all OOCE forms must be turned into QA. Copies must be included in each SDG or workorder in validatable packages and in the first workorder in the "samples affected" column for non-validatable data packages.

3.4.9 Any corrective actions taken which could either impact data directly, help to explain analytical decisions that were made in order to resolve analytical discrepancies, or which would help in the interpretation of the final data package must also be narrated in the final report. OOCE forms must be turned in with the data and the supervisor creating the narrative comment for that area will comment on any decisions resulting from failed QC which could impact data validity or interpretation.

Appendix I

Corrective Action Form

Laucks Testing Laboratories
Corrective Action Report

1) Problem Description:

Response tasked to: ______________________ on ______________________

By: ______________________ Response Requested By ______________________

2) Cause:

3) Action Taken:

Completed by ______________________ on ______________________

- ☐ Corrective actions will be reviewed 30 days after completion to verify problem has been corrected.
- ☐ No further action necessary

Reviewed by: ______________________ on ______________________

1) Person initiating corrective action fill out Part 1 and may fill out Part 2 if they are aware of the cause
2) Original goes to person tasked with a response: one copy goes to QA Officer and another kept by person initiating corrective action
3) Person tasked completes response in Part 2 (if not previously completed) and Part 3, signs response, and returns original to person initiating action
4) Person initiating action determines if action corrects the problem and signs "Reviewed by." If action was insufficient, return to the person charged with responding without signing.
5) Completed original goes to QA Officer

Appendix 2

QC_DB Report Form

Laucks Testing Laboratories

QC_DB Report Form

Analyst________

Checker________

Test Code________

		QC Exceeds Control Limit √ if yes	Corrective Action Approved By ____
PBlk	B______96______	☐	
MS/MSD	K______96______	☐	
SRM	R______96______	☐	
Blk Spk	S______96______	☐	
MS/Dup	M______96______	☐	
Duplicate	D______96______	☐	

This report validates the following work orders ________________________________

Appendix 3

Out-Of-Control Event Form

OUT OF CONTR__ EVENT FORM

No:______________

Date Recognized: ______________________
Date Occurred: ______________________
Method: ______________________
Initiated By: ______________________
Analyst: ______________________

- ☐ GC/MS VOA
- ☐ GC/MS ABN
- ☐ GC VOA
- ☐ GC non-VOA
- ☐ HPLC
- ☐ Metals
- ☐ Wet Chemistry
- ☐ Extractions
- ☐ Data Management

Samples Affected (Workorder & Sample Numbers.)

Type of Event: (*check all that apply*)

___ Holding time missed (*describe below*)
___ Blank ≥ MDL___ RL___ CRQ/DL___
___ Spike Recoveries do not meet criteria
___ Duplicate RPDs do not meet criteria
___ MS/MSD Results do not meet criteria ___ %Rec ___ RPD
___ BS/BSD Results do not meet criteria ___ %Rec ___ RPD
___ Analytical Spike recoveries do not meet criteria
___ Standard Additions do not meet criteria
___ LCS or Blank Spike Recoveries do not meet criteria
___ Surrogate Recoveries do not meet criteria
___ Calibration Corr. Coefficient does not meet criteria
___ Calibration Verification does not meet criteria ___ Init ___ Cont.
___ Recovery ___ Retention time ___ %D
___ Tuning fails criteria
___ ISTD fails criteria
___ Calculation/Transcription error
___ Other (explain)

____
____
____
____

Corrective Action: (*check all that apply*)

___ Repeat Calibration
___ Made new standards
___ Reanalyzed, Date: ____________
___ Sample(s) Redigested/Reextracted Date: ____________
___ Results Recalculated
___ Cleaned System
___ Ran Standard Additions
___ Notified Client ______________________
___ Other (Please explain)

____
____
____
____

Check One:

___ Original Results Reported

___ Rerun Results Reported

Notified:

___ QA

___ Client Services

Action taken By: ______________________ Date: ____________ Reviewed by Initiator: ______________________ Date: ____________

___ Out of Control Event Corrected By: ____
____
____
____

___ Corrective Actions Not Successful (signatures required) **DATA MUST BE FLAGGED AND/OR NARRATED**

Analyst: ______________________ Date: ______________________
Supervisor: ______________________ Date: ______________________

Distribution:
Original to QA
Copy to workorder / SDG file for all validatable packages and to

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike Program**

Revision history:

Number	Date
LTL-0048 Rev1.1	05/18/92
2	06/21/96

Written by: Harry Romberg Date: 6-21-96
Harry Romberg, QA Officer

Approved by: Karen J Kotz Date: 6/21/96
Karen Kotz, Laboratory Director

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Table of Contents

1. Introduction and Scope

1.1 Description

1.1.1 This SOP provides a description of how blind spikes are generated, what types of analyses are monitored, how results are evaluated and how Laucks handles out of specification events.

1.1.2 Materials may be from a multitude of sources. The analyst will most often be aware that the sample is a blind spike but in no case should the analyst know the "true" value of the submitted sample. On occasion, at the discretion of QA, a double blind sample may be submitted (one which the analyst does not know is an evaluation sample).

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Definition of Terms

1.2.1 **Blind Spike** - A proficiency sample which may or may not be known as such by the analyst but which contains a target analyte with a value which is not known.

1.2.2 **Double-Blind Spike** - A proficiency sample which is submitted to the analyst in such a way that it is thought to be a routine sample and which contains an unknown amount of target analyte.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Pipets. flasks, containers etc. necessary to prepare spikes for submission.

2.2 Reagents

2.2.1 Deionized water, methylene chloride and other solvents or preservatives that may be required to prepare spikes. Some samples may be prepared by outside sources and only need to be submitted to the analyst.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances. During the preparation of blind spikes, the analyst will be exposed to a variety of reagent chemicals and solvents. In addition, preservatives contained in both reference materials and in sample bottles may pose health hazards. The health effects of these various chemicals may be ascertained by reading the appropriate material safety data sheets (MSDS). It is incumbent on the analyst to exercise due care and caution while executing this SOP. The company will provide any protective equipment or clothing needed to assure employee safety.

3.1.2 Many solvents also pose a fire hazard and should be treated with proper precaution.

3.2 Waste Disposal

3.2.1 Waste solvents are disposed in the appropriate waste solvent container.

3.2.2 No more blind spike material is used than is necessary for submittal of the sample so th it will not present a disposal hazard.

3.2.3 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on hazardous waster disposal.

4. Materials

4.1 Sources

4.1.1 Materials may be WS, WP or other materials from an external performance evaluation. Although these are not generated directly by the laboratory, they are blind samples in that the expected values and in many cases the constituents themselves are not known to the analyst beforehand.

4.1.2 Standard materials may be purchased from a vendor, such as Environmental Resource Associates (ERA), Analytical Products Group (APG), SPEX, Restek, Supelco or any other reputable vendor.

4.1.3 Materials may be purchased either as Performance Evaluation samples (values unknown to the laboratory), reference materials (values known to the laboratory), or as standard materials (values known to the laboratory). They may also be made up by supervisory QA staff from materials of known content. In any instance, the value of the componen will be unknown to the analyst performing the analysis until completion of the evaluation.

4.2 Storage

4.2.1 Materials are stored as recommended by the manufacturer, most often at a temperature of 4°C ± 2°C.. Metals will generally be stored in dilute nitric acid and need not be refrigerated.

5. Operation procedures

5.1 Requirements and Scheduling

5.1.1 These requirements may be program and/or method-specific. Laucks specific training requirements and documentation are discussed in other SOPs and in the QA Plan. This SOP is intended primarily to document the practices and evaluation of results and not to dictate the specific analyst requirements.

5.1.2 Initially (as part of being considered able to independently perform an analysis), an analyst may be required to analyze a single blind Performance Evaluation (PE) sample. The analyst must process the samples independently, without direction or assistance in order to be considered proficient.

5.1.3 On an ongoing basis, at least annually, an analyst may also be required to demonstrate continuing performance by analyzing a single blind PE sample.

5.1.4 PE results may also be used as a supplement to a method verification process in order to verify the laboratory's ability to perform a method.

5.1.5 These PE samples may be from a performance evaluation study, such as an EPA Water Pollution (WP) or Water Supply (WS) study, an independent vendor PE, such as Environmental Resource Associates (ERA) or Analytical Products Group (APG), or it may be prepared by an area supervisor from a known material. Blind PE samples will almost always be prepared as aqueous solutions except in limited circumstances, such as fuel hydrocarbons, where soil samples are periodically analyzed. ERA, APG or other sources of materials will be used where components are not present in WP, WS or other "official" PE samples. Acceptable results from programmatic samples, such as those for HAZWRAP, Army Corps of Engineers, or NFESC may be used to qualify analysts or to otherwise demonstrate performance, even though in some instances an actual value may not be provided by the agency.

5.1.6 WP and WS program samples are analyzed semiannually (WP in approximately June and November, WS in approximately April and September). Supplementary PE samples for analytes not present in these samples (such as fuels or GC/MS semivolatiles) are generally obtained from APG, ERA or a similar vendor and are generally analyzed along with remedial samples (if any) resulting from WP failures (results being obtained approximately 3 months after submittal of the WPs). Other external PE samples from programs such as NFESC, HAZWRAP, or the Army Corps of Engineers may be analyzed at the discretion of those programs but be used for evaluation. The precise schedule for submittal of all but programmatic samples is at the discretion of QA in order to meet laboratory needs to qualify analysts or methods or to meet other requirements.

5.1.7 One set of PE samples may be used to qualify several analytical staff. For instance, one person may extract a sample and be so qualified. Several analysts may process the extract independently and also be qualified. If multiple analysts do process the extract, however, there must be no collaboration between analysts until the results have been received by QA.

5.1.8 In any instance, the values of the components must not be divulged to the analyst(s) prior to analysis. Furthermore, if a PE sample contains one or more components from a multi-component analysis (such as a semivolatiles or pesticide mixture), the analytes themselves must not be divulged.

5.1.9 Blind spikes should be analyzed in at least duplicate so that reproducibility can be determined as well as recovery. All results should be reported for each determination where the analysis was otherwise in control. Evaluation of replicates is a laboratory option and is rarely required of any external performance evaluation program.

5.1.10 Blind spikes are typically determined for the following analyses (in water excepts as noted):

- ICP metals
- ICP/MS metals
- Graphite furnace metals (Pb, As, Se, Tl)
- Mercury
- GC Volatiles
- Gas/BTEX water & soil
- Diesel water & soil
- Petroleum Hydrocarbons (418.1) water & soil
- Pesticides
- GC/MS Volatiles
- GC/MS Semivolatiles

- PNAs
- Explosives
- Cyanide
- Total Organic Halogens
- Total Organic Carbon
- Phenolics
- Ion Chromatography (F, Cl, NO_3, SO_4)
- NO_3/NO_2 Automated Cd reduction
- others at the discretion of QA

5.1.11 Where other method references are very similar to those above, the same PE analysis may be considered adequate documentation for both methods. Other blind PE studies may be conducted at the discretion of QA.

5.1.12 Samples will be given a laboratory ID number and test code when they are submitted to the laboratory and should be tracked in the same manner as a routine sample. Results will be compared against vendor-supplied, method-specific, or laboratory-derived limits as noted in the Evaluation and Reporting section.

6. Evaluation and Reporting

6.1 Data Package Organization

6.1.1 Paperwork must be completed as it would for routine samples, documenting preparation, calibration, and analysis and quality control. In addition, a summary page must be completed with the results of the sample and any replicate analysis. The summary page must contain the following elements:

- Analyst
- Date of analysis
- Preparation Technician (where appropriate)
- Date Prepared
- Analysis (Method*)
- Preparation (Method*)
- Components obtained from the analysis
- Results obtained from the analysis
- Replicates (where applicable) and associated RPDs

* At the discretion of QA, analysis and preparation methods may be considered sufficiently similar to qualify for more than one reference technique.

6.2 Evaluation

6.2.1 The data will be evaluated by QA with possible assistance from other supervisory staff. Data must meet the limits supplied by the vendor, if purchased or supplied as part of a PE program. If limits are not given by the vendor, method specific limits may be adopted or the laboratory may choose to accept recoveries based on internal QC limits.

6.2.1.1 All relevant components must be identified by the analyst, although in a few limited cases, similar components react in much the same fashion (i.e. similar retention times or patterns). In these instances, at the discretion of QA, the analyst may be allowed to re-evaluate the analysis.

6.2.1.2 If the analysis is a multi-component mixture, the results may be considered acceptable if 90% of the target analytes are quantified correctly.

6.2.1.3 Replicates will most often be evaluated where recovery exceptions occur or where it is determined by QA or the area supervisor that this reproducibility is a critical part of the analyst's evaluation. They will also be evaluated if it is so specified in the reference method. In these instances, the acceptability criteria are generally either the laboratory-derived RPD(s) or the reference method-specified criteria.

6.2.1.4 At the discretion of QA, the data may also be evaluated for completeness and documentation.

6.3 Remedial Actions

6.3.1 If the limits for the analyzed material have been exceeded, that performance criterion will be considered to have not been met. In such case, the data will first be re-evaluated by the analyst. If sufficient extract/digestate remains, this may include re-analysis.

6.3.2 If, after re-evaluation, the performance criterion still has not been met, the results from the entire analysis will be evaluated and if sufficient criteria have not been met, the analyst may be required to analyze another blind PE sample.

6.3.2.1 In some cases, the quality of the vendor-supplied material may be in question. In this instance or in the case where no more of a specific material is available in a timely fashion, a second source of performance evaluation material may be used.

6.3.3 Continued failure may result in either or both examining the analysis/preparation method for discrepancies or it may require re-training of the analyst if it is determined that the method and instrumentation is functioning properly. In either case, action must be initiated immediately to insure that accurate results are being produced for actual laboratory samples.

6.3.4 In the extreme case, it may be determined after consultation with supervisory staff and laboratory management (including QA), that no analyses can be performed using that method or that analyst until there is demonstration of adequate performance.

7. Record Keeping

7.1 Analyst and Method

7.1.1 Records for all evaluations will be maintained by QA. Analyst evaluation will be maintained in the analyst's training file. Method evaluations will be kept separately but may mirror the analyst's evaluation.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1011

Title: **Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits**

Revision history:

Number	Date
4	1/30/98
3	6/21/96
2	1/08/96
1.1	6/15/90

Revised by: Harry Romberg
Harry Romberg, QA Officer
Date: 1-30-98

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director
Date: 1/30/98

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Table of Contents

1. Introduction and Scope

1.1 Overview

1.1.1 This SOP describes the determination of Instrument Detection Limits (IDLs), Method Detection Limits (MDLs), Precision and Accuracy Studies, the setting of Reporting Limits and the determination and use of control limits. All are defined in the definitions section of this SOP.

1.1.2 In general, detection limits are the minimum amount of a target analyte that can be measured and determined to be greater than zero with a known degree of confidence. For purposes of this SOP, the known degree of confidence for MDLs will be defined as the 99% level. IDLs are based strictly on instrument response and MDLs on a sample processed through the entire preparation process. This SOP is based on information provided in 40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11* and in other sources such as the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) and SW846.

1.1.3 Criteria for Precision and Accuracy (P&A) Studies are generally defined in the specific published method, particularly those in SW 846. Where criteria are not so defined, Laucks has chosen to either use the criteria from similar methods or to set in-house criteria based on the judgment of senior management and QA. Where two methods are the same in technical detail and one does not provide P&A criteria, performance under the guidance of the method with specifications may be used to satisfy the performance criteria of both.

1.1.4 Control limits are determined initially for an analysis, generally using limits supplied in the method or defined by the program (such as CLP). After sufficient points have been accumulated the laboratory performs a statistical analysis of the data and computes the control limits which are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

1.1.5 This SOP is designed for applicability to a wide variety of sample types ranging from reagent water to solids containing the analyte. The MDL may vary as a function of sample type. Laucks rarely determines MDLs on any matrix other than soil or water. Other MDLs may be estimated based on these studies.

1.1.6 This SOP requires that a specific, detailed analytical method exist. When determining MDLs and P&As following this SOP, it is imperative that all sample processing steps included in the analytical method be included.

1.1.7 Where a specific method has requirements exceeding the requirements of this SOP, that method will take precedence. Where a reference method has stated detection limits, these are generally taken to be MDLs. This SOP is to be followed to validate a new method or to validate a change in a current method.

1.1.8 MDLs should be determined approximately annually for common procedures and as needed for procedures which may be performed on an infrequent basis.

1.1.9 PCB MDLs are to be performed for each PCB to be analyzed. At least one PCB MDL must be determined annually and all PCB MDL determinations must be performed within 3 years.

1.1.10 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis except in the case of P&A studies which are used to demonstrate the competency of the analyst.

1.2 Method Description

1.2.1 Detection Limits

1.2.1.1 For any metals method, the Instrument Detection Limit (IDL) must first be determined. The IDL may also be determined strictly for informational purposes for other methods but is not required. The IDL allows the analyst to assess the precision of the measurement system and to estimate the target concentration for the MDL study. IDLs are generally determined by analyzing 7 low-level standard replicates on 3 non-consecutive days and averaging the sample standard deviations from each of the three days.

1.2.1.2 In order to determine MDLs, a minimum of seven replicate measurements are made of a prepared sample matrix which contains approximately 1 to 5 times the estimated detection limit. A Student's t determination is made for the number of data points available, usually 7 (6 degrees of freedom), and the resulting standard deviation multiplied by that value to determine the MDL. All MDL data are entered into the laboratory MDL database.

Note: The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Laucks considers up to 10 times the MDL to be an appropriate concentration although limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

1.2.1.3 Reporting Limits (RLs) are set by the laboratory as limits that can be reliably reported on a consistent basis with a reasonable degree of confidence that the reported level is accurate. These limits may be set at the Practical Quantitation Limit (PQL) initially by using a multiplier times the MDL. The multiplier is often but not always defined in the method. After initial setting of the RL, it is rarely changed unless significant changes in the MDL occur which make it necessary to raise or lower the RL.

1.2.2 Precision and Accuracy (P&A) Studies are studies performed in order to demonstrate the laboratory's ability to perform a method and are also used to demonstrate analyst competency to perform the method. They generally involve the analysis of 4 replicates spiked at concentrations defined in the method. Where no method guidance is provided, the replicates should be prepared at concentrations of 10 to 50 times the MDL for each analyte. Adequate performance is most often defined in the reference method, although if the method performance has been demonstrated, analyst competency may be demonstrated in comparison to laboratory limits.

1.2.3 Control limits may be specified in a reference method or may be statistically determined by the laboratory from existing data. In general, laboratory determined limits for control samples must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory cannot demonstrate adequate performance for all samples on a routine basis.

1.2.4 It is not uncommon for clients to specify reporting or control limits in their project quality assurance plans. As long as they are achievable (i.e. the requested RL is not lower than the laboratory MDL), Laucks will generally comply with the client's request for that particular project.

1.3 Definition of Terms

1.3.1 **Accuracy** - The degree of agreement of a measurement (of an average of measurements of the same thing), X, with an accepted reference or "true" value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100*(X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in the system. Accuracy shall be calculated as follows:

$$\%R = \frac{C_s - C_u}{S} * 100$$

Where:

Cs = Concentration of spiked sample
Cu = Concentration of unspiked sample
S = Expected concentration of spike in sample
%R = Percent recovery

1.3.2 **Control Limits** - Control limits may be specified in a reference Method (either as mandatory or guidance limits), or may be developed by the laboratory using internal performance data. Control limits represent acceptance criteria for determining whether an analytical system is in control (functioning within acceptable guidelines).

1.3.3 **Control Sample** - A QC sample introduced into the analytical process to allow evaluation of the measurement system. In general, it is best to use samples of a matrix similar to the samples being analyzed, where such are available. The control sample, however, will generally be free from interferences other than those inherent to the matrix itself.

1.3.4 **Degrees of Freedom** - The number of independent estimates that could be obtained from a specific set of data. In general, for a simple set of *n* independent values,

$$df = n-1$$

1.3.5 **IDL** - Instrument detection limit - The lowest concentration of a target analyte that can be measured and known to be greater than the instrumental background with a known degree of confidence. It may be used as a starting point for selecting MDL study spiking levels.

1.3.6 **MDL** - Method detection limit - The minimum concentration of a substance that can be measured and reported with a known degree of confidence (99% for our purposes) that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

1.3.7 **Mean** - The arithmetic sum of a set of observations divided by the number of observations.

$$\frac{\sum X_i}{n}$$

Where:
X_i = sample value for replicate i
n is the number of replicates

1.3.8 **P & A** - Precision and Accuracy - This often refers to a study conducted to validate a method or an analyst conducting a particular method.

1.3.9 **PQL** - Practical Quantitation Limit - The limit at which it is determined that the constituent can not only be detected but be accurately quantified. This limit is usually 2 to 10 times the MDL but may be even larger depending upon the constituent and the matrix. Factors are often taken from the published method but may be set by the laboratory if published factors do not exist. These limits may also be used as the routine reporting limit (RL), unless otherwise contractually defined.

1.3.10 **Precision** - A measure of mutual agreement between individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".

1.3.11 **Reporting Limit (RL)** - A value greater than or equal to the MDL or the IDL which may be based on QA decision, the published method specifications, or project-specific requirements.

1.3.12 **Standard deviation** - A statistical measure of the variability of a set of sample observations. For the purposes of this SOP, the sample standard deviation is used. This is calculated using the formula:

$$s = \sqrt{\frac{\sum \left(X_i - \overline{X}\right)^2}{n-1}}$$

Where:
s = the standard deviation estimated with n-1 degrees of freedom.
X_i = sample value for replicate i
$\overline{X}$ = mean of all of the replicates
n = the number of replicates

2. Equipment List and Standards

2.1 Equipment, Reagents and Standards

2.1.1 As appropriate for the given analysis.

2.1.2 Personal Computer with access to a spreadsheet program such as *Microsoft Excel* and the laboratory MDL database.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Refer to the specific analytical SOP for appropriate safety precautions.

3.1.2 Waste Disposal

Refer to the specific analytical SOP for appropriate waste disposal practices. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1.1 Calibration is as appropriate to the specific method. No matrix spiking or other routine QA is required.

5. Responsibilities

5.1 Analyst

5.1.1 Each analyst is responsible for verifying a valid MDL study was performed and is available for each method they perform. In addition, each organic instrument analyst is responsible for verifying a valid annual MDL was performed on each instrument for each method they perform.

5.1.2 Each analyst is responsible for producing a one-time initial demonstration of precision and accuracy.

5.1.3 A metals analyst is responsible for assuring that a quarterly IDL study is produced on each instrument.

5.1.4 Each analyst is responsible for labeling MDL and P&A studies appropriately.

5.1.5 Each analyst is responsible for turning in a legible MDL, IDL, and P&A study to their supervisor for review and approval prior to final submittal to QA.

5.1.6 All of the analyst activities should be coordinated through the area supervisor.

5.2 Supervisor or Senior Analyst

5.2.1 Each area supervisor or senior analyst is responsible for coordinating the effective completion of the required studies. This may include but not necessarily be limited to helping determine appropriate concentration levels, coordinating the completion of the study within the timeline required by the method and/or the QA department, and scheduling the study around the analytical workload.

5.2.2 It is the responsibility of the area supervisor or senior analyst to insure that the analyst is performing the study within the guidelines of the method and to perform a review of the final data prior to submission to QA. This review should include determination that appropriate spiking levels were used, that the data was properly computed and transcribed, and that any problems or concerns encountered during the study are documented. Part of this review must include the comparison of the data to method specific criteria. In other words, P&A data must be compared to established method criteria and MDLs must be compared to Reporting Limits to ensure they are no greater than the RLs.

5.2.3 It is the responsibility of the area supervisor to obtain the necessary information to update the control limits at a minimum of annually. This may be done in conjunction with QA and the LIMS/MIS department.

5.3 QA Department

5.3.1 It is the responsibility of the QA department to issue a Corrective Action notice to any department who fails to turn in acceptable MDL, IDL, or P&A studies.

5.3.2 It is the responsibility of the QA department to work with supervisors to schedule studies and to maintain files of all current and historical studies.

5.3.3 QA will review and provide the final sign-off that the study meets requirements.

5.3.4 QA will review and provide the final sign-off of reporting limits.

5.3.5 QA will bear the responsibility to maintain the statistically determined control limits and to ensure that they are within those specified in the reference method.

6. Operation procedures

6.1 General

6.1.1 All studies must be given laboratory LIMS ID numbers. Although they may be initially stored in QA, they will eventually be moved into the laboratory filing system and must have identification numbers in order to be able to retrieve the raw data. Identification numbers will almost always be assigned by QA but in the absence of the QA Officer may be assigned by authorization of QA or the Laboratory Director. All studies will use the SAM client code QC_Officer in order to better track them at a later date.

6.2 Instrumental Detection Limits (IDLs)

6.2.1 It is rarely necessary to perform actual IDL studies except for metals analyses. For metals analyses, they are performed quarterly on each instrument. Studies may be useful, however, to demonstrate instrument capabilities and as a tool for estimating the Method Detection Limit (MDL). Although IDLs may be used as estimates to determine appropriate MDL spiking levels, it is strictly prohibited to compute the actual MDLs based on IDL determinations. The following guidelines are provided for several general class of analyses, regardless of whether an IDL is required for that analysis type.

6.2.2 As with all studies, a laboratory ID number should be assigned by QA for tracking purposes. In the case of metals IDLs, the same ID number may be assigned to all of the quarterly IDLs, rather than just one per instrument.

6.2.3 Actual IDLs studies are performed according to the CLP SOW by analyzing 7 replicates of low-level standards made up in the same matrix as all standards and not including any processing steps that would not ordinarily be performed on standards. The levels of those standards should be estimated from manufacturers detection limit specifications.

6.2.4 IDLs should be performed under the same instrumental conditions as will be used to perform actual analyses.

6.2.5 IDL studies must contain the following information (not necessarily in this order) for submittal to QA.

- Laboratory ID number
- Analyst who performed the IDL study
- Instrument name and ID which will distinctly identify that instrument
- Spike level
- Measured concentration of the 7 replicates (per day)
- Standard Deviation

- Mean
- Determined IDL
- Concentration Units
- Date(s) the study was analyzed
- Analysis (i.e. ICP, GFAA, etc.)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.2.6 Spectrophotometry

6.2.6.1 The EPA/CLP SOW for metals requires that the IDL study be run on 3 non-consecutive days at least 7 times each day. It is prepared from an acidified aqueous standard solution made up at 3 to 5 times the manufacturers suggested IDL. The sample standard deviation (n-1) for each individual set of determinations is calculated and the final IDL is calculated as 3 times the average of the standard deviations for the three days. This may be performed using any commercial spreadsheet but care must be taken to insure that it is done using the sample standard deviation (n-1) calculation. For *Microsoft Excel*, this is the =STDEV() calculation. Ten percent of the calculations must be manually verified in order to demonstrate that the spreadsheet calculations are accurate.

6.2.6.2 If other spectrophotometric method IDLs are established by analyzing standards 7 times on 3 non-consecutive days, the calculation of the IDL is performed as described above. In addition, the EPA/CLP method does not prescribe the determination of MDLs. It is standard laboratory procedure to perform an MDL study (see section 6.3) approximately annually for almost all routine methods of analysis, regardless of IDL frequency or other determinations.

6.2.7 Chromatography

6.2.7.1 The analyst should use the signal:noise method for determining concentrations to use for an IDL study. A preliminary estimate of 5x signal:noise is to be used; if necessary this will be adjusted and the study repeated.

6.2.8 Gas Chromatography/Mass Spectrophotometry

6.2.8.1 Mass spectral identification criteria are key in selecting target concentrations for the IDL study. The mass spectroscopist's experience in determining the minimum identifiable concentration must weigh heavily in selecting concentrations. All compounds must meet the spectral matching characteristics as called out in the analytical method for the IDL study to be valid.

6.3 Method Detection Limits (MDLs)

6.3.1 MDL studies must be performed annually for each method for inorganic analysis and for each method/instrument combination that will be used for organic methods.

6.3.2 MDL studies must also be performed when any major changes have been made in an instrument, such as a detector change.

6.3.3 Prior to beginning an MDL study, a laboratory workorder ID must be obtained from QA. The data generated from the study is then referenced to that workorder in the same manner as routine sample data.

6.3.4 MDL studies must contain the following information (not necessarily in this order). This will be accomplished by using the MDL database report plus an MDL Information Sheet (See Appendix 2).

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation (where applicable)
- Date(s) the study was prepared
- Method number of the clean-up (where applicable)
- Analyst who performed the MDL study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 7 replicates
- Standard Deviation
- Mean
- Determined MDL
- Concentration Units
- Reporting Limits (RLs)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.3.5 The analyst must compare the MDLs with their current Reporting Limits (RLs) to ensure that they are no higher than the RLs. In fact, in most cases the MDLs should be demonstrably lower than the RLs unless there is a specific request to report down to the MDL.

6.3.6 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL and cannot be re-determined with more appropriate results), the QA Officer and the supervisor, often in concert with the Laboratory and/or Technical Director(s), must meet to determine the appropriate course of action. Reporting limits are intended to be at a level for which method precision and accuracy can be obtained. This generally cannot be done when the RL is close to the MDL.

6.3.7 In order to determine the Method Detection Limit (MDL), it is first necessary to estimate what the MDL will be in order that the appropriate spiking levels may be used. How this estimate is made is immaterial to the actual MDL determination. Methods for making this determination may include any one or a combination of the following:

- estimating based on the instrument detection limit (IDL) as determined above or by any other means
- estimating based on the previous MDL
- estimating based on 3 times the instrument signal to noise ratio
- estimating based on analyst judgment

6.3.8 A solution is then prepared and spiked into a sample matrix, which is as free as possible of interference and target analytes, at a level that will result in a sample concentration equivalent to 1 to 5 times the estimated MDL.

> **Note:** The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Although the analyst should make his/her best effort to spike at a level from 1 to 5 times the MDL, Laucks considers up to 10 times the MDL to be a sufficient concentration. Limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

6.3.8.1 Spiking levels which are determined to be less than 1x or greater than 10x the MDLs should in almost all circumstances be re-analyzed at a more appropriate spiking level.

6.3.8.2 Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interference concentrations are not detected at the estimated method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species. The interference concentration is presupposed to be normally distributed in representative samples of a given matrix.

6.3.9 Preparation of Spiked Samples

6.3.9.1 The MDL is almost always determined in reagent water or clean sand. Prepare a laboratory standard containing all analytes of interest at a concentration which is at least equal to or in the same concentration range as the estimated MDL. The analyte concentration should not exceed 5x the estimated MDL but allowances may be made up to 10x the determined MDL.

6.3.9.2 It is extremely rare that Laucks will perform studies for other than reagent water or soil. Soil matrix will almost always be represented by clean blank sand except for metals analyses where even clean sand contains levels of some metals which exceed the 10x acceptance criteria. For such analyses, reagent spikes are used containing only the digestion/preparation reagents. MDLs on other matrices will generally only be performed upon specific client request.

6.3.10 Calculation of recovery statistics

> **Note:** All values are used without correcting for native concentration. As previously mentioned, if blank correction is a part of the method, the average blank value is used for correcting analyte concentration measurements. In almost all methods, however, blank correction is forbidden.

6.3.10.1 The sample standard deviation is calculated as follows:

$$s = \sqrt{\frac{\sum (X_i - \overline{X})^2}{n-1}}$$

where:
s is the standard deviation estimated with n-1 degrees of freedom.
X_i = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
n is the number of replicates

6.3.10.2 The Student's *t* statistic is determined for (*n* -1) degrees of freedom at the 99% confidence interval (CI). A Student's *t* table for the 99% CI is provided in Appendix 1. For most data sets, using n=7 sample readings, the *t* value is 3.143.

> **Note:** In some cases, it may be determined that it is useful to prepare an additional sample so that, in case of laboratory accident, at least 7 are available for statistical analysis. Whether or not this is done, all samples analyzed **must** be used in the statistical evaluation unless there is a strong reason to reject one or more of the data sets, such as obvious contamination, abnormally poor surrogate recovery, set of values that are in obvious and significant disagreement with all of the others, or spilled sample. It is inappropriate to reject data which do not have an overriding reason to do so. The reason for rejection must be clearly documented in the data file. If more than 7 points are used in the MDL determination, the current MDL database will not accommodate the calculation. In this case, the determinations will necessarily be done using a spreadsheet program.

6.3.10.3 The MDL determination then becomes:

$$MDL = t_{99\%CI} * s$$

where:
$t_{99\%CI}$ = the Student's t value at the 99% confidence interval
s = the sample standard deviation as calculated above

6.3.10.4 The MDL, standard deviation and Student's t statistic for the appropriate number of replicates at the 99% CI are automatically calculated when using the Laucks MDL database.

6.3.11 Methodology Exceptions/Specifics

6.3.11.1 Wet Chemistry

6.3.11.2 The MDL for all titrimetric determinations is set as the value determined by 0.2 ml of titrant at the method specified titrant strength and sample aliquot size. This would include all tests such as versenate hardness, alkalinity, argentometric or mercurimetric chloride, titrimetric COD, etc. Karl-Fisher moistures would be an exception to this; the MDL is taken to the value determined by 0.05 ml of titrant, the method specified titrant strength, and sample size.

6.3.11.2.1 The MDL for all gravimetric residue determinations (total solids, total suspended solids, etc.) is set as the value determined by a weighing of 0.2 mg at the method specified sample size.

laboratory may be demonstrating adequate performance on the control material in any specific analytical run, it cannot demonstrate adequate performance for all samples in that run on a routine basis.

6.6.7 The laboratory may also calculate limits for matrix spike and matrix spike duplicate or replicate samples. However, these limits are primarily used to demonstrate method performance on a particular sample or sample-type relative to the routine laboratory sample and exceptions to these limits will generally be allowed as long as control sample limits are met.

6.6.8 The laboratory may be called upon to utilize control limits specified in a method or in a specific contract as designated in the LIMS ProjQC database or supplementary paperwork. The laboratory's overall performance will be considered adequate if internal control limits are within those specified in the reference method. Contractually defined limits will be used for the control samples analyzed under the contract and appropriate corrective actions taken but will not be used as a guide for routine laboratory performance.

6.6.9 For any particular project, if the laboratory exhibits exceptions to the method or contract-specified criteria, appropriate corrective action must be taken. Should routine laborato control limits be within method or contract-specified criteria, and laboratory limits are exceeded but method or contract limits are met, the data may be reported but should be flagged. Where appropriate, corrective action may still be taken at the discretion of QA.

7. Reports

7.1 Data Package Organization

7.1.1 All work, with the exception of control limit computations, is performed under laboratory workorder ID numbers.

7.1.2 All data supporting the study are provided in a standard format specific to that method. In order to save paper, some items, such as the initial calibration, etc., may be referenced to other workorders. However, it must all be easily recoverable if full documentation is required, up until the standard laboratory data disposal date. Rationalizations for interpreting the results of any study and specific detail which might impact the study should be documented in the file as well.

7.1.2.1 Data files are prefaced with a copy of the summary report containing all of the elements previously noted in this SOP. Where laboratory database reports are available, a copy of the database report must also kept on file by QA. All sign-offs will be handwritten.

6.4.4 Project Specific RLs are derived from project requirements and are contractually agreed upon between the laboratory and the client. In any event, the agreed upon limits **cannot** be less than the MDL or IDL.

6.4.5 On occasion, the low standard defines the RL. The decision to use this technique may be any combination of method specific requirements, laboratory decision, or project-specific requirements. In no case will the RL determined from the low standard be lower than the statistically determined MDL.

6.4.6 Reporting Limits are generally detailed in the Detection Limits Database and the LIMS system, unless set by project-specific agreement, in which case they are detailed in documents pertaining to that project and in the ProjQC database. The only persons given the capability to edit the approved limits are QA, LIMS system administrators, and the Technical or Laboratory Director. In most cases, only QA will actually perform any such editing. Note here that the EPA Contract Laboratory Program (CLP) requirements use specific contract required detection limits (CRDLs) or quantitation limits (CRQLs) and any project using the CLP methods will almost always also be reported using the CLP CRDLs or CRQLs. Any exception to the use of the CLP limits in these instances must also be noted in the ProjQC database and on any paperwork defining the details of the project.

6.5 Precision and Accuracy Studies

6.5.1 At a minimum, a one-time demonstration of precision and accuracy (P&A) must be performed for each method.

6.5.2 In some cases, it may also be required that an analyst will be required to perform a P&A study to be considered proficient and capable of independently performing a preparation or analysis.

6.5.3 P&A studies will be performed in accordance with the specific method. Where method-specific performance criteria are not specified, Laucks may choose to set criteria independently. Laucks' criteria, at a minimum, will meet those specified in a given method. Any determination to the contrary must be well documented and in direct consultation with QA and laboratory management.

6.5.4 All P&A studies must be turned in to QA after having undergone supervisory or senior analyst review.

6.5.5 All P&A studies must include the following information:

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation
- Date(s) the study was prepared
- Analyst who performed the analysis portion of the P&A study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 4 replicates
- Standard Deviation of the recovery tabulated against the published QA Acceptance Criteria Table, where available
- Average recovery tabulated against the published QA Acceptance Criteria Table
- Concentration Units
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed
- Raw Data

6.5.6 The mean recovery and acceptance limits must meet the criteria given in the QC Acceptance Criteria Table at the end of each of the determinative methods, when available. Where criteria are not available Laucks may use internal acceptance criteria or defer to a similar technical method with P&A criteria and use this P&A criteria as guidance in establishing performance criteria. In the case of organic SW846 methods, if the criteria are not published in the individual method, the criteria in method 8000 (70%-130%) are followed as a guidance. In many instances, 70-130% is not achievable on a routine basis even by skilled staff. In this case, the laboratory (senior staff in conjunction with QA) may determine its own acceptance limits.

6.5.7 Blank spike analyses are the commonly accepted P&A evaluation. In most methods where criteria are defined, 4 replicates must meet method-specified criteria for the **laboratory** to be considered capable of adequate performance.

6.5.8 The individual **analyst** must be able to analyze four replicates and meet laboratory blank spike control limits to be considered competent to perform the applicable analysis. For purposes of the P&A study, the analyst may be considered qualified if 90% of the analytes in a multi-analyte analysis meet laboratory criteria as long as all analytes meet the default method-specific criteria.

6.5.9 For the laboratory to be able to claim routine performance within specified limits. all analysts performing an analysis must be capable of that level of performance. All analysts must be routinely capable of performance within method-specified criteria and will be evaluated against laboratory criteria, with further action and training in order if they are unable to routinely meet laboratory criteria.

6.6 Control Limits

6.6.1 Initially, when a new method is being implemented or there are insufficient data, the laboratory will use method-specified control limits for evaluation of data. If no such limits exist, the laboratory may elect to use specified limits from a similar method or may set default limits at the laboratory's discretion. These limits may be from the precision and accuracy study for that method. The determination for the suitability of setting any default limits not otherwise specified in a reference method is at the discretion of QA.

6.6.2 During the routine course of analysis, blank spike or laboratory control samples (LCS) and in many cases matrix spikes and matrix spike duplicates (or sample duplicates) will be analyzed. Spiking will occur at the levels specified in the respective methods where available, but will generally be somewhere in the middle of the calibration range.

6.6.3 When sufficient data have been gathered, generally at least 20 data points, the laboratory will undertake the determination of statistically-based control limits. These control limits are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

6.6.4 At a minimum, the control limits will be updated annually on a preparation/analysis/matrix specific basis. The number of data points and spiking levels used to obtain the new limits must be documented when forwarded to QA for approval.

6.6.5 If purchased from a commercial vendor, vendor-supplied control limits for a control sample will be considered adequate for default control limits if they are within the limits specified in the reference method. In addition, if the material is readily available and its composition does not change with every purchase, the laboratory will develop internal limits for that material. These limits may or may not be within the vendor-supplied limits but they **must** be within the method-specified limits.

6.6.6 In general, laboratory determined limits for **control samples** must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because even though the laboratory may be demonstrating adequate performance on the control material in any specific analytical run, it cannot demonstrate adequate performance for all samples in that run on a routine basis.

6.6.7 The laboratory may also calculate limits for matrix spike and matrix spike duplicate or replicate samples. However, these limits are primarily used to demonstrate method performance on a particular sample or sample-type relative to the routine laboratory sample and exceptions to these limits will generally be allowed as long as control sample limits are met.

6.6.8 The laboratory may be called upon to utilize control limits specified in a method or in a specific contract as designated in the LIMS ProjQC database or supplementary paperwork. The laboratory's overall performance will be considered adequate if internal control limits are within those specified in the reference method. Contractually defined limits will be used for the control samples analyzed under the contract and appropriate corrective actions taken but will not be used as a guide for routine laboratory performance.

6.6.9 For any particular project, if the laboratory exhibits exceptions to the method or contract-specified criteria, appropriate corrective action must be taken. Should routine laboratory control limits be within method or contract-specified criteria, and laboratory limits are exceeded but method or contract limits are met, the data may be reported but should be flagged. Where appropriate, corrective action may still be taken at the discretion of QA.

7. Reports

7.1 Data Package Organization

7.1.1 All work, with the exception of control limit computations, is performed under laboratory workorder ID numbers.

7.1.2 All data supporting the study are provided in a standard format specific to that method. In order to save paper, some items, such as the initial calibration, etc., may be referenced to other workorders. However, it must all be easily recoverable if full documentation is required, up until the standard laboratory data disposal date. Rationalizations for interpreting the results of any study and specific detail which might impact the study should be documented in the file as well.

7.1.2.1 Data files are prefaced with a copy of the summary report containing all of the elements previously noted in this SOP. Where laboratory database reports are available, a copy of the database report must also kept on file by QA. All sign-offs will be handwritten.

8. References

40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11*

EPA Contract Laboratory Program (CLP) Inorganics Statement of Work (SOW), ILM04.0.

EPA "600" Series Methods, section 8.1.1, 40 CFR, Part 136, App. A.

EPA SW846 "8000" Series Methods, both the general method 8000B and the specific methods

Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center, February 1996

Appendix I

Student's *t* Values

n	degrees of freedom	*t* value at 99% CI
2	1	31.821
3	2	6.965
4	3	4.541
5	4	3.747
6	5	3.365
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567
19	18	2.552
20	19	2.539
21	20	2.528
22	21	2.518
23	22	2.508
24	23	2.500
25	24	2.492

Appendix II

MDL Information Sheet

Laucks Testing Laboratories, Inc.
MDL Information Sheet

This form must be submitted in addition to the information supplied on the SAM MDL summary form along with the supporting raw data.

SAM MDL Name: ____________________

SAM Workorder Number: ________________________

Analysis:

Analyst: ____

Analysis Method: ________________________

Analysis Date: _________________________

Instrument ID: _________________________

Preparation:

Prepared by: ____

Preparation Method: ________________________

Preparation Date: _________________________

Cleanup Method(s): ________________________

Review:

Analyst's Signature: ____________________________________

Supervisor's Signature: ___________________________________

QA Approval: ____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1012

Title: Solvent QC Monitoring for Trace Residue Analysis

Revision history:

Number	Date
2	4/27/99
1	4/10/91

Written by: Harry Romberg   Date: 4-29-99
Harry Romberg, Quality Assurance Officer

Reviewed by: Monica Carr   Date: 4/29/99
Monica Carr, Organics Division Manager

Approved by: Kathy Kreps   Date: 4/29/99
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope

1.1 Description

1.1.1 The purpose of this SOP is to define the method(s) used to check and document the purity of the major solvents used for trace residue analysis at Laucks. The solvents being tested are methylene chloride, acetone, and hexane. Specific techniques and equipment used for operations such as concentration and solvent exchange are not addressed in this document.

2. Equipment List and Reagents

2.1 Equipment and Reagents

2.1.1 Other Glassware, reagents and equipment as delineated in the methods specific to the described task.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Typical precautions should be taken when handling any solvent. Some, such as methylene chloride or freon are not flammable, but others, such as acetone and hexane are and should be treated with extreme caution. Long term health effects of solvent contact are often unknown but generally unfavorable. Breathing of ANY solvent vapor should be minimized, as should any direct skin contact, by working in a well ventilated area (in or near a hood if necessary) and by using the provided gloves and, if necessary or desired, respirator masks.

3.2 Waste Disposal

3.2.1 All waste solvents should be disposed in the appropriate waste solvent container, never in the sink or in combination with aqueous liquids.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on hazardous waste disposal.

4. Operation procedures

4.1 Sequestering of Solvent

4.1.1 A new lot of acetone or methylene chloride must be sequestered by the supplier and the checking process initiated at least four weeks prior to using up the last of the previous lot of that solvent. A lot is defined as a batch of solvent with the same manufacturers lot number. This must be done in order to ensure that the lot has been released for analytical use BEFORE the remainder of an acceptable lot has been used up. If any solvent has failed, a second bottle may be tested for the failed parameter(s) in order to ensure the failure was not due to laboratory contamination. Failure of the second test is grounds to reject that lot for use in the laboratory.

4.1.2 When a lot has been formally designated as acceptable, enough should be ordered to last approximately 2 months in order to minimize the frequency of testing necessary. Typically, the supplier will ship 10 cases of a sequestered lot of methylene chloride each week and acetone as requested. No more than 4 months supply of methylene chloride will ever be ordered, as typical methylene chloride recommended shelf-life is 6 months.

4.1.3 Alternatively, since it is unlikely that any lot will fail and to eliminate the time between acceptance and delivery, an appropriate supply (as defined above) may be ordered and sequestered at the laboratory for analysis. This is commonly done for hexane since we do not consume large quantities of this solvent. If said lot fails, however, the lot must be returned to the supplier and a new lot tested immediately. This lot MUST be kept separate from the current stock and very clearly marked so that it is not inadvertently used prior to acceptance. This distinction is the responsibility of the Extractions Supervisor. All solvent deliveries must be immediately reported to the Extractions Supervisor or designated alternate in order that this distinction be made.

4.2 Initiation, Data Handling and Record Maintenance

4.2.1 The Extractions supervisor or designated representative initiates the checking process. For any month in which any extractions solvent or other reagent QC is performed, a LIMS workorder is created. When a bottle from a new, previously untested lot of solvent is received, a sample ID number is assigned from the next available ID in the workorder. This "fraction" ID must contain the manufacturer, lot number, solvent, and tests (test codes) to be performed.

4.2.2 The three solvents which we specifically check in the extractions lab are methylene chloride, acetone, and hexane. The test codes used for these purposes are MECLAC, MSQCCK, and PXQCCK. These codes are used <u>only</u> for testing solvents and for nothing else. Other checks, such as sodium sulfate or Florisil should use the test code appropriate for the analysis for which the material will be used (8081, 8270, etc.)

4.2.2.1 Methylene chloride is checked for acidity, semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are MECLAC, MSQCCK, and PXQCCK.

4.2.2.2 Acetone is checked for semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are MSQCCK, and PXQCCK.

4.2.2.3 Hexane is checked only for pesticides. The respective test code assigned to this analysis is PXQCCK.

4.2.3 When testing has been completed, the lot will be officially designated as acceptable or failed by the QA Officer or Technical Director. This will be done by initialing the final report and sending a copy to the Extractions supervisor. Any lot will be considered acceptable which meets the criteria specified in Appendix I. The Extractions supervisor should be certain that a lot has been designated as acceptable prior to using it and should take whatever actions are necessary to ensure prompt analysis and acceptance before the last of the acceptable solvent has been used.

4.2.4 The data and report files will be maintained by the QA Officer. After all of the QC in the month has been closed and signed off, the data file and acceptance sheets will be filed with the regular workorder files.

4.3 Solvent Analysis

4.3.1 Methylene Chloride Acidity

4.3.1.1 0.01 N NaOH - To a 100 mL. volumetric flask, add 10 mls. of .1000 N sodium hydroxide from the buret of standardized NaOH in the Inorganics lab. Fill to the volumetric mark with deionized water, stopper, and mix very well. It takes several inversions of the flask to properly mix the solution (at least 10). This solution should be prepared immediately prior to analysis.

4.3.1.2 Neutral ethanol - Add 25 mls. of denatured ethanol to an Erlenmeyer flask. Add 2 or more drops of phenolphthalein indicator solution (1 gm. phenolphthalein/100 mls. ethanol). With a Pasteur pipet, add the .01 N NaOH solution dropwise until the ethanol turns slightly pink. Hold the flask against a white background to enhance the color. This solution should be prepared immediately prior to analysis

4.3.1.3 Add 25 mls. of the methylene chloride to be checked to the flask containing neutralized ethanol. Swirl. Do not shake too vigorously so that CO_2 from the air will not acidify the ethanol and cause a fading endpoint.

4.3.1.4 Add 900 uL of the 0.01 N NaOH. Swirl to mix well.

4.3.1.5 If the resulting color is pink, the methylene chloride passes (is not acidic). If it does not turn pink, it should be retested, preferably from a second bottle. If it fails a second time, it should be rejected or used only for cleaning. Failing solvent should NEVER be used for extraction purposes.

4.3.1.6 A "PASS" or "FAIL" is entered into the SAM report under the associated regular SAM test code, MECLAC. If the solvent fails, residue analysis SHOULD NOT be performed until a suitable acceptable lot is determined. The Extractions supervisor should see that any such failing lot has been terminated in SAM. Data and the report, however, should still be submitted to the QA Officer.

4.3.2 Residue Checks

4.3.2.1 The residue checks are performed for EPA CLP Target Compound List (TCL) components for both pesticides/PCBs and semivolatiles (ABNs) as is appropriate for the solvent being checked.

4.3.2.2 In all cases, varying amounts of the appropriate solvent is concentrated to 1 mL. in a Kuderna-Danish concentrator. No splitting of the concentrate occurs. Surrogates are not added.

4.3.3 Methylene Chloride - $MeCl_2$ is used for both ABN and pesticide/PCB analyses. A separate 400 mL. concentration is done for each analysis.

4.3.3.1 For the pesticide/PCB analysis, hexane is added and the solvent exchanged and concentrated down to 1.0 mL., which is submitted for analysis.

4.3.3.2 For ABN analysis nothing is added and the $MeCl_2$ concentrated directly down to 1 mL and submitted for analysis.

4.3.4 Acetone - Acetone is used for both ABN and pesticide/PCB analyses. A separate 150 mL. concentration is done for each analysis.

4.3.4.1 For the pesticide/PCB analysis, hexane is added and the solvent exchanged and concentrated down to 1.0 mL., which is submitted for analysis.

4.3.4.2 For the ABN analysis, the acetone is blown down to near dryness with nitrogen and brought up to 1 mL. with $MeCl_2$ and submitted for analysis.

4.3.5 Hexane - Hexane is used only in pesticide analysis. It will be concentrated 25 mls. to 1 mL. as stated and submitted for TCL pesticide/PCB analysis.

4.3.6 Acceptance criteria are compiled in Appendix I and are based on the appropriate amounts of solvent concentrated to 1 mL. final volume. They are derived from Laucks reporting limit criteria for acceptable blanks. The LIMS report indicates the acceptance level, the level found and signifies whether the detected level (if any) passes (OK) or fails (FAIL).

4.4 Data Package Organization

4.4.1 A copy of the signed acceptance form along with the raw data is retained by QA under the assigned laboratory number. The Extractions supervisor keeps a photocopy of the signed approval sheet in a file in the extractions lab.

Appendix I

Solvent Acceptance Criteria

Solvent Acceptance Criteria

Semivolatile Compounds	Total ng in 1 mL.
Phenol	5000
bis(2-Chloroethyl) ether	5000
2-Chlorophenol	10000
1,3-Dichlorobenzene	5000
1,4-Dichlorobenzene	5000
1,2-Dichlorobenzene	5000
2-Methylphenol	5000
2,2'oxybis(1-Chloropropane)	5000
4-Methylphenol	5000
N-Nitroso-di-n-propylamine	5000
Hexachloroethane	5000
Nitrobenzene	5000
Isophorone	5000
2-Nitrophenol	10000
2,4-Dimethylphenol	5000
bis(2-Chloroethoxy)methane	5000
2,4-Dichlorophenol	10000
1,2,4-Trichlorobenzene	5000
Naphthalene	5000
4-Chloroaniline	5000
Hexachlorobutadiene	5000
4-Chloro-3-methylphenol	5000
2-Methylnaphthalene	5000
Hexachlorocyclopentadiene	5000
2,4,6-Trichlorophenol	10000
2,4,5-Trichlorophenol	10000
2-Chloronaphthalene	5000
2-Nitroaniline	5000
Dimethylphthalate	25000
Acenaphthylene	5000
2,6-Dinitrotoluene	5000
3-Nitroaniline	5000
Acenaphthene	5000
2,4-Dinitrophenol	10000
4-Nitrophenol	10000
Dibenzofuran	5000
2,4-Dinitrotoluene	5000
Diethylphthalate	25000

Semivolatile Compounds	Total ng in 1 mL.
4-Chlorophenyl-phenyl ether	5000
Fluorene	5000
4-Nitroaniline	10000
4,6-Dinitro-2-methylphenol	10000
N-nitrosodiphenylamine	5000
4-Bromophenyl-phenylether	5000
Hexachlorobenzene	5000
Pentachlorophenol	10000
Phenanthrene	5000
Anthracene	5000
Carbazole	5000
Di-n-butylphthalate	25000
Fluoranthene	5000
Pyrene	5000
Butylbenzylphthalate	25000
3,3'-Dichlorobenzidine	10000
Benzo(a)anthracene	5000
Chrysene	5000
bis(2-Ethylhexyl)phthalate	25000
Di-n-Octylphthalate	25000
Benzo(b)fluoranthene	5000
Benzo(k)fluoranthene	5000
Benzo(a)pyrene	5000
Indendo(1,2,3-cd)pyrene	5000
Dibenz(a,h)anthracene	5000
Benzo(g,h,i)perylene	5000

Solvent Acceptance Criteria

Pesticide/PCB Compounds	Total ng in 1 mL.
alpha-BHC	5
beta-BHC	5
delta-BHC	5
gamma-BHC	5
Heptachlor	5
Aldrin	5
Heptachlor epoxide	5
Endosulfan I	5
Dieldrin	10
4,4'-DDE	10
Endrin	10
Endosulfan II	10
4,4'-DDD	10
Endosulfan sulfate	10
4,4'-DDT	10
Methoxychlor	50
Endrin ketone	10
Endrin aldehyde	10
alpha-Chlordane	5
gamma-Chlordane	5
Toxaphene	500
Aroclor-1016	100
Aroclor-1221	200
Aroclor-1232	100
Aroclor-1242	100
Aroclor-1248	100
Aroclor-1254	100
Aroclor-1260	100

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1013

Title: **Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials**

Revision history:

Number	Date
1	8/31/92
2	4/17/96
3	6/3/96

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 6-5-96

Reviewed by: Monica Carr
Monica Carr, Organics Manager
Date: 6/6/96

Reviewed by: Bill Lundberg
Bill Lundberg, Inorganics Manager
Date: 6/6/96

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director
Date: 6/6/96

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Table of Contents

1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP is intended to describe the way in which standards and reference materials are tracked, prepared, stored and maintained at Laucks, from the time of receipt of the neat or stock materials, solutions or their preparation to the point of use of the working standard. General descriptions of documentation of standard preparation may be present, it is not intended to define the actual method of preparation for each specific method. This is contained in the applicable analytical method SOP. The way in which these standards are tracked, however, is detailed along with the description of storage and shelf life guidance.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described procedure of documentation.

1.2 Definition of Terms

1.2.1 Standard or Reference Material: these items are defined as any solution of an analyte at a known concentration prepared from purchased neat materials or stock solutions, or from intermediate solutions traceable to purchased materials. This includes calibration standards, independent laboratory control standards (LCS or SRM), spiking solutions, surrogate solutions, independent calibration verification standards.

2. Equipment Lists and Standards

2.1 Equipment

2.1.1 Equipment and reagents necessary for the preparation of any specific solution.

2.2 Standards

2.2.1 Standards as specified in each analytical SOP.

2.2.2 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by either (1) purchasing solutions which have been fully documented by a commercial vendor, or (2) following the recommended steps for traceability as outlined in the 3/90 CLP Organic statement of work.

2.3 Standards Logbooks

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards and reference materials including neats or solutions should be handled as if they are hazardous substances.

3.2 Waste Disposal

3.2.1 Waste segregation and disposal from the point of collection is further covered in Laucks SOP on Hazardous Waste Disposal.

4. Operation Procedures

4.1 Preparation of Organics and Inorganics Materials

4.1.1 General consideration in standard preparations include:

4.1.1.1 Determine volumes and aliquots required using the concentration calculations in Appendix 1.

4.1.1.2 Choose volumes and aliquots which minimize the number of intermediate dilutions required to obtain final working concentration considering:

- The inherent measurement error, i.e. no aliquots less than 20% of the volume of measurement device whenever possible.
- The ratio of solvent:analyte
- The amount of solution left over for disposal.

4.1.1.3 Be sure to use a solvent volume sufficient to dissolve all analytes.

4.1.1.4 The solvent used should be miscible with water when being used for sample spiking purposes. Most standards used in the extractions laboratory are prepared with methanol.

4.1.2 Proper Syringe/Pipette Technique

4.1.2.1 Choose an appropriate size syringe so that the measured volume is at least 2/3 of the total volume of the measurement device.

4.1.2.2 When selecting a pipette, choose volumetric pipettes only for the exact amount to be measured.

4.1.2.3 Always rinse a syringe (organics) at least ten times with the appropriate solvent in between measurements, and wipe the syringe with a Kim-wipe.

4.1.2.4 There should be no air bubbles. Either tap them away or discard the solution in the syringe/pipette and obtain another aliquot. Repeat this procedure as often as necessary to remove all bubbles. It may be helpful to use a GC septum with very small (<50 µl) syringes.

4.1.2.5 For organics, when delivering the measured volume to the dilution vessel, fill the vessel 1/2 - 2/3 with the solvent to be used, add the measured aliquot directly into the solvent without touching the sides of the container, and fill to volume with solvent. A sub-surface injection is preferable whenever possible.

4.1.3 When preparing stock solutions from neats, the following steps should be taken.

NOTE: 99.9% of the time, stock standards will be prepared WEIGHT per Volume. DO NOT use Volume measurements for liquids unless EXPRESSLY TOLD to do so by your SUPERVISOR.

4.1.3.1 The dilution vessel (volumetric flask) and stopper should be triple solvent rinsed (last time with the solvent to be used for standard preparation) and allowed to dry completely.

4.1.3.2 The neat is weighed, to 4 significant figures, directly into the volumetric flask and the weight is recorded (to 3 decimal places for volatiles, one less than actually weighed in order to account for possible small losses due to volatilization). Stopper before weighing to avoid compound volatilization if dealing with solvents or volatile materials.

4.1.3.3 For components other than volatiles, the volumetric flask is filled about 3/4 full with dilution solvent and shaken until analyte is completely in solution.

- If the analyte will not dissolve, the stoppered volumetric flask should be sonicated in the sonic bath until it does dissolve. (Because sonication heats the solution slightly, the solution should be allowed to cool before dilution to the mark). Consult your supervisor if the compound is not in solution after sonication.

- The volumetric flask is diluted to the mark.
- If the analyte recrystallizes while stored in the refrigerator, the standard should be sonicated before use. Do not aliquot from a cloudy or opaque standard.
- In addition to the normal labeling of the standard, a separate label should be added indicating the need for sonication.

4.1.3.4 For volatiles, the flask is inverted and gently mixed three times after diluting to the mark.

4.2 Traceability Documentation for Organics and Inorganics Materials

4.2.1 All organic neat standard materials are logged into the NEATS database, as described in 4.2.5, when they arrive in the lab. No neat organic material should be used before it has been logged into the database. Inorganic stock materials are logged directly into the appropriate standards logbook. Examples of some NEATS database screens are provided in Appendix 3.

4.2.2 All standard, spike, or surrogate mixes which are diluted solutions, whether organic or inorganic in nature, are not logged into the database but are logged directly into the appropriate standards logbook.

4.2.3 The current controlled logbooks are identified in each area as follows:

- GC/MS Volatiles - MV# (used for standards made from neat materials, single analyte concentrates, or supplier provided standard mixes)
- GC/MS Semivolatiles - MS#
- Metals - ME#
- GC Pesticides - PX#
- GC Volatiles - VOA#
- GC & HPLC PNAs - BA#
- other GC & HPLC analyses - MA#
- Organic Extractions misc - EX#
- Technicon & Lachat Analyzers - TE#
- IR Oil and Grease - IN#

- Ion Chromatography - IC#
- TOC/TOX - OC#

NOTE 1: # in the above table indicates a sequential number, beginning with 1, with each subsequent controlled book with that analysis code having the next higher integral value.

NOTE 2: This logbook number is for tracking standards only. The logbooks also will have a QA logbook number used for controlling logbooks which is independent of the standards tracking process.

4.2.4 All purchased stocks and subsequent standard preparations must be recorded in the appropriate database or log-book.

4.2.5 Upon receipt, each purchased neat material, stock, intermediate or working solution is entered into either the database (if an organic neat material) or a standards log-book and assigned a unique LAUCKS identification number. The information entered in the database or standards logbook must include:

- Analyte(s) name and vendor product ID (vendor ID must be given to unequivocally identify exactly what was used).
- supplier name
- supplier lot number
- concentration and/or purity
- expiration date (either vendor supplied, the analytical SOP or determined from the shelf life table in Appendix 2, in order of preference)

NOTE: In the case of the metals solutions which are supplied without an expiration date, the date opened and corresponding expiration date will be added when the standard is opened based on, in order of preference, the analytical SOP or Shelf Life table in Appendix 2.

4.2.6 After each material is logged it is labeled with the LAUCKS ID, date received, date opened (if the material is to be used from the same container more than once) and expiration date (if not already on the label). The accompanying vendor Certificates of Analysis, Purity or Authenticity are labeled with the Laucks ID and filed in a controlled laboratory notebook in the laboratory area. These certificates are then archived through QA when the notebook is full.

4.2.7 Every prepared stock, intermediate or working standard solution is entered into the standard log-book and assigned a unique LAUCKS ID number. The logbook entry must include the items detailed in section 4.2.9. Each material must be labeled with LAUCKS ID number, preparation date, expiration date and preparer's initials. Other items to be included on the label are listed in section 4.3.1. Examples of typical standards logbook entries are provided in Appendix 4.

4.2.8 An example of the solution nomenclature used is a working ABN standard prepared on 11/13/91. The solution number assigned was MS 2-77-2. This label represents the following:

- MS - solution was made and used as a semivolatile mass spec standard
- 2- solution was logged into standard book #2
- 77- page number on which solution has been recorded
- 2- this denotes the second entry on page 77

4.2.9 All discrete measurements made during a standard preparation must be recorded in the log book, specifically, weights aliquots and final volumes.

Other pertinent data to be entered in the log book are as follows:

- Standard Name
- Parent material and concentration/purity
- Solvent/Diluent standard is prepared in
- Type of standard being prepared (i.e. inter-mediate, spike, working, calibration)
- Final concentration
- Date prepared/opened
- Expiration dates
- Analysts initials

4.2.10 The Laucks internal working material ID must be documented on the manual benchsheet the analytical run-log or instrument printout to enable tracking back to the parent material. See Appendix 5 for examples of typical bench sheets with standards references.

4.3 Storage of Standards and Reference Materials

4.3.1 Always completely label solution with the following information:

- LAUCKS ID number
- Standard name
- Concentration
- Solvent/Diluent
- Technician's initials
- Date of preparation
- Expiration Date

4.3.2 Organic Standards and References Materials

4.3.2.1 Store in vial or bottle which minimizes head space.

4.3.2.2 Use amber or clear glass, screw tops with Teflon-liners when required, and store at, in order of preference, the temperature referenced in the analytical SOP or the temperature detailed below, in the assigned refrigerator.

4.3.2.3 Volatile Standards and Reference Materials

4.3.2.3.1 All standards solutions should be stored in the VOA freezer at -10°C to -20°C.

4.3.2.3.2 Most volatile standards are stored in the original ampules until used.

4.3.2.3.3 Standards are transferred to Mininert vials with Teflon lined septa for daily use and stored in the VOA freezer. When the standards are transferred, the information is recorded in the GC/MS Volatile Standards log book.

4.3.2.4 Other Volatile Standard Solutions

4.3.2.4.1 Some standards need to be prepared in the lab. Stock solutions are diluted using high purity MeOH.

4.3.2.4.2 To insure stability, standard solutions should be sealed in amber glass ampules

4.3.2.4.3 Rinse unsealed ampules with clean MeOH and place in oven to dry.

4.3.2.4.4 Cover ends of ampules with foil.

4.3.2.4.5 Dilute stock solution in high purity MeOH in a volumetric flask.

4.3.2.4.6 Mix gently.

4.3.2.4.7 Partially fill ampules with solution and recap with foil.

4.3.2.4.8 Use CO_2 to cool ampules until crystals form on sides.

4.3.2.4.9 Heat end of ampule with acetylene flame until glass begins to soften.

4.3.2.4.10 Gently pull end until seal is formed.

4.3.2.4.11 Label ampules and store in freezer.

4.3.2.4.12 Record the information in the Mass Spec VOA Standards Log Book (MV).

4.3.2.4.13 When standard solutions are used they should be transferred to Mininert cap vials with Teflon lined septa. The vials are stored in the VOA freezer until discarded.

4.3.2.5 Semivolatile Standards and Reference Materials

4.3.2.5.1 All standards solutions should be stored at a maximum temperature of 4 degrees C (± 2 degrees). Refer to the analytical SOPs for details as some analytes may drop out of solution if at cooler temperatures.

4.3.3 Inorganic Standards and Reference Materials

4.3.3.1 All metals standards are kept in a cabinet in the metals analysis lab. This is at room temperature. Expired standards that are kept for qualitative purposes are kept in the same room, in a different cabinet. These qualitative standards have a special label on the bottles denoting that they are not to be used for quantitative purposes. All other standards are kept at 4°C in a reach-in cooler in the inorganics lab. This cooler is dedicated to standards and SRMs only. No sample storage is allowed in this cooler.

4.4 Shelf Life

4.4.1 Expiration

4.4.1.1 If a parent material has an expiration date of month/year, then the material is considered usable through the end of that month. For example, 01/96, the material expires after 1/31/96. This guidance was obtained from various vendors.

4.4.1.2 All parent expiration dates MUST be entered into the standard log books and the expiration date for all resulting child materials must also be entered into the logbook and placed on the material label.

4.4.1.3 Note that no child solution may exceed the life of a parent solution or neat material This stipulation may reduce the shelf life of a prepared solution from that listed in Appendix 2. For instance, if a stock solution is prepared from parent material that has an expiration date of 05/20/95 in 01/95, instead of having a six month shelf life (07/95) the solution will expire, 05/20/95, the same date as the parent.

4.4.1.4 See Appendix 2 for the Table of typical shelf life of standards and reference materials. This table is provided as guidance only. The vendor expiration date (if applicable) and the analytical SOP take precedence over any guidance set forth in the Table.

4.4.1.5 If a standard is past its expiration date it may be used for qualitative purposes only. The standards logbook must be edited to reflect this status and an additional label must be placed on the standard. This label must be bright in color and must indicate that it is to be "Used for Qualitative Purposes Only".

5. Standard Verification

5.1 Criteria

5.1.1 Standards are to have their concentrations verified before use whenever possible. The QC'ing of the standard is to be recorded in the applicable column in the standards logbook unless they are validated in the individual analytical run (such as confirmation by another standard from an independent source). Criteria for standards acceptability are in many cases defined in individual SOPs. In instances where they are not so defined, acceptability criteria are:

- 80% - 120% for organics
- 90% - 110% for inorganics

Appendix 1

Example Calculations

1. Concentration Calculations from Neat Materials

HELPFUL hint: To keep yourself straight ALWAYS, ALWAYS include the units (mg, ml, etc.) in your calculations.

Example Calculations of Standard Concentrations:

Weight of Neat Material:	0.2500 gm
Volume of Solvent:	10 ml

To Calculate Concentration in mg/L (ppm):

1) Calculation in Steps.

A) $0.2500gm * \frac{1000mg}{1.0g} = 250mg$

A.1) Adjust the 250 mg for purity,

i.e. if purity = 90%, 250 mg x 0.9 = 225 mg

B $10mls * \frac{1L}{1000mls} = 0.01L$

C) $\frac{225mg}{0.01L} = 22500mg/L$

2) Calculation as a Single Step.

$$\frac{0.2500gm}{10ml} * 0.90(purity) * \frac{1000mg}{1gm} * \frac{1000ml}{1L} = 22500mg/L$$

3)

$$FC = \frac{W}{FV} * P * \text{Conversion Factors}$$

where;

W = Weight of neat material (g)
FV = Final Volume (ml)
P = Purity (%/100)
FC = Final Concentration (mg/L = ppm)

2. Intermediate and Working Standards (Standard Dilution)

$$(FC)(FV) \times 1000 = (AV)(PC)$$

where;

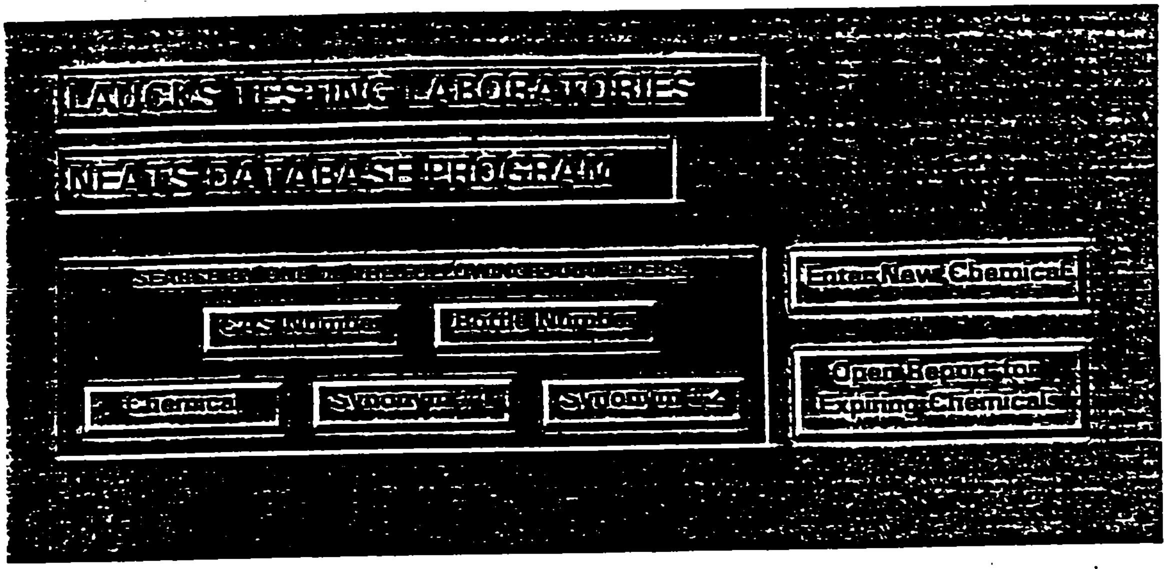
FC: Final Concentration(s) in standard desired. Units=µg/mL.
FV: Final volume of the prepared standard. Units=mL.
1000: Conversion factor from mL to µL
PC: Parent Concentration (standard normally containing high concentrations and is diluted to desired final concentration). Units = µg/mL.
AV: Aliquot Volume of parent standard required to achieve final concentrations desired.
Units: µL (microliter).

a) Neats to Stocks

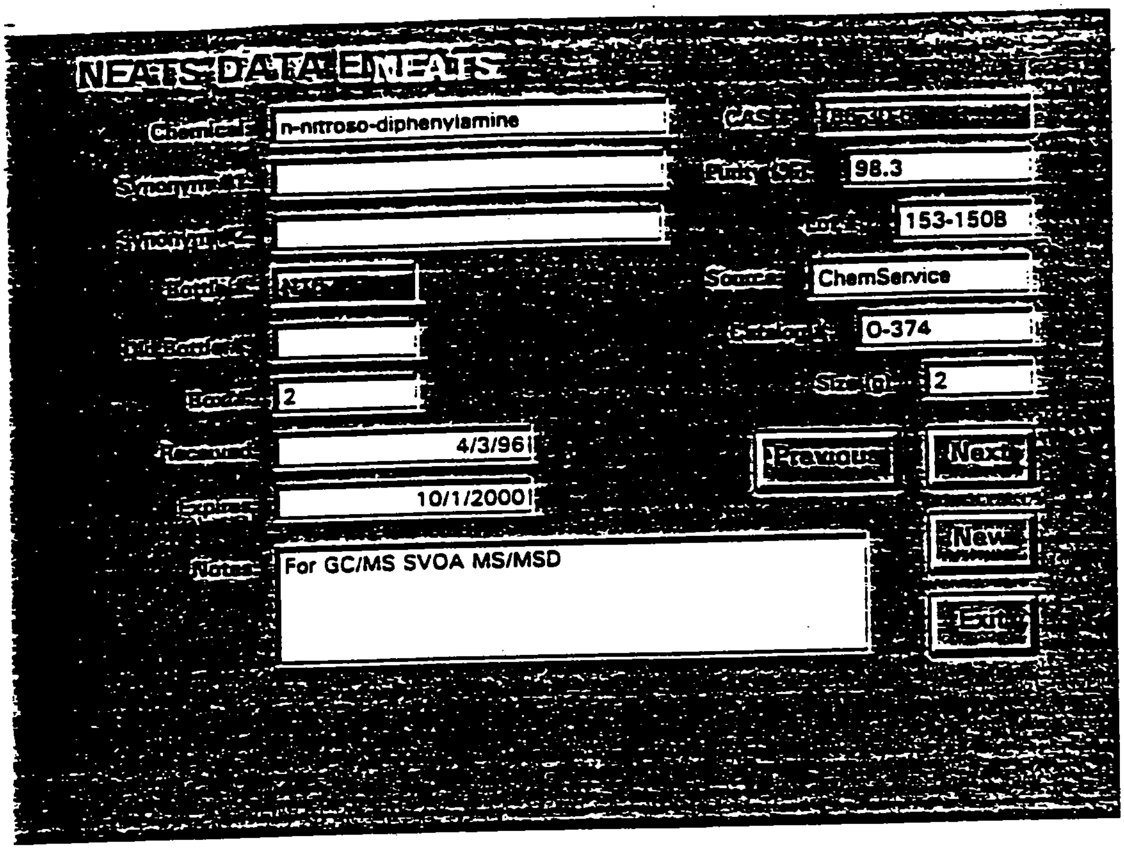
$$\frac{Purity * 1{,}000{,}000 * W}{FV} = FC$$

where;

1,000,000 = Conversion factor from gram to microgram
W Weight used in standard prep (g)
FV Final Volume (ml)
FC µg/ml = ppm = mg/L
Purity = % Purity/100



LAUCKS TESTING LABORATORIES
NEATS DATABASE PROGRAM
CAS Number
Bottle Number
Chemical
Enter New Chemical
Open Report for
Expiring Chemicals


Chemical
n-nitroso-diphenylamine
CAS
Purity (%)
98.3
Lot
153-150B
Source
ChemService
Catalog
O-374
Size (g)
2
Box
2
Received
4/3/96
Expires
10/1/2000
Notes
For GC/MS SVOA MS/MSD
Previous
Next
New
Exit

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

MH4-03-04 con't

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
4	2,4,6-TNT	M-8330-11	1000 PPM	10 ml	50 ml	200 ppb	DI H_2O	12/20/95	MH	04/01/96	
	HMX	M-8330-4									
01	1,3,5-TNB	M-8330-12	1000 PPM	10 ml	50 ml	200 PPb	Fluid TCLP #1	12/20/95	MH	04/01/96	
	2,4-DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6-TNT	M-8330-11									
	HMX	M-8330-4									
02	1,3,5-TNB	M-8330-12					TCLP #2 Fluid				
	2,4,-DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6-TNT	M-8330-11									

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	IC ICV Lot [illegible] Cl	ERA 34112	—	—	250ml	129 mg/L	—	logged in 3/18/96	SK	Opened: 4/9/96 Exp: 4/9/97	
	F		—	—		6.47 mg/L	—				
	NO_3		—	—		8.77 mg/L	—				
	SO_4		—	—		110 mg/L	—				
2	Cal. Std. F	IC4.84.4	1000γ	500µl	100ml	5γ	H_2O	3/20/96	SK	4/3/96	
	Cl	.5	1000γ	1000µl		10γ					
	NO_2	.7	100γ	2000µl		2γ					
	NO_3	.3	1000γ	500µl		5γ ~~10γ~~ SK 3/20/96					
	SO_4	.6	1000γ	2000µl		20γ					
3	NO_2 ICV	IC4.78.7	100γ	1000µl	100ml	1γ	H_2O	3/21/96	SK	3/27/96	
4	NO_2 ICV	IC4.78.7	100γ	1000µl	100ml	1γ	H_2O	3/25/96	SK	4/1/96	

Appendix 5

Bench Sheet Examples

As, Tl

AutoSampler Report   Table: SW846   Wed 04-10-96 09:39:45 AM   page

...le Name: SW846   Autosampler Type: TYPE TJA
...le Positions: 257/300   QC Positions: 11/19   # Sets: 1
Rinse Station location is rack -1, pos. -1.

--- Racks ---

Rack #	Type	Usage	#Pos Left	Analyses/Pos
1	Aux. (L) Rack	STD/QC/BLANK	11	10
2	Sample (13mm)	Samples	32	1
3	Sample (13mm)	Samples	75	1
4	Sample (13mm)	Samples	75	1
5	Sample (13mm)	Samples	75	1

--- Sample Sets ---

Set#	Type	Prepare?	Description	Method	#Pos	Rack#	Start
1	,Y	No	UMC09,UMC11 RE-AS,TL	UMASOIL	43	2	1

Report As, Tl
KP 4/10/96

--- Preparation Info ---

Set#	Uptake	Uptake#2	Final	Dil.Factor

No Samples Prepared.

: #1

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	ICV1 ME4-48-01	-NA-	1	QC Standard
2	1	2	STD4 ME4-48-01	-NA-	1	Standard
3	1	3	STD3 ME4-48-01 5mls/10mls	-NA-	1	Standard
4	1	4	STD2 ME4-48-01 1ml/10mls	-NA-	1	Standard
5	1	5	STD1 ME4-45-02	-NA-	1	Standard
6	1	6	STD0	-NA-	7	Blank
7	1	7	Blank	-NA-	6	QC Standard
8	1	8	CCV ME4-51-01			

(9...19 Not Used)

Rack #2

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	CRII1 ME4-45-02	1	-NA-	Sample
2	1	2	ICSABI1 ME4-52-05	1	-NA-	Sample
3	1	3	PBS1	1	-NA-	Sample
4	1	4	LCSS1	1	-NA-	Sample
5	1	5	03040-01	1	-NA-	Sample
6	1	6	03040-01D	1	-NA-	Sample
7	1	7	03040-01S	1	-NA-	Sample
8	1	8	03040-01L	1	-NA-	Sample
9	1	9	03040-02	1	-NA-	Sample
10	1	10	03040-03	1	-NA-	Sample
11	1	11	03040-01 5X	1	-NA-	Sample
12	1	12	03040-01D 5X	1	-NA-	Sample
13	1	13	03040-01S 5X			

LAUCKS TESTING LABORATORIES
ABN GC/MS OPERATIONS LOG

CASE # ____________

PAGE 4883-A

3/18/96 IS = MS334-1 DFTPP = MS334-4

RUN #	FILE NAME	CLIENT NAME	CLIENT SAMPLE#	LAB I.D. #	INJECTION INFO	IS A RT	IS A RESPONSE	IS B RT	IS B RESPONSE	IS C RT	IS C RESPONSE
1	MT7501										
2	HC18A	—	SSTD050	MS4-9-1	25. Y ABN	11.24	55075	14.99	159300	20.44	104485
3	HC18B	—	SSTD200	MS3-87-1	100. Y Nonyl phenol	11.22	59100	14.98	169403	20.42	100469
4	HC180	Kalama	B031396MSWLA	9603368-Blk	1000→1	11.23	58139	14.98	166207	20.43	99304
5	HC181	Kalama	S031396MSWLA	-SPK	1000→1	11.24	55723	14.99	155289	20.44	98643
6	HC182	Olympia	—	9603206-01DL	1:4 1000→1	11.24	52364	14.98	158592	20.44	102071
7	HC183	Kalama	—	9603368-01	1:2 100→1	11.24	56997	14.98	168568	20.46	103091
8	HC184	↓	—	-01DL	1:4 100→1	11.24	58386	~~14.~~ 15.00	173480	20.45	106213

JJ 3/19/96

LAUCKS TESTING LABORATORIES INC.
Seattle. Washington

SOP #:LTL-1017

Title: Internal Audit Procedures

Revision history:

Number	Date
000	5/13/93
001	3/3/96

Written by: Harry Romberg   Date: 3/3/96
Harry Romberg

Approved by: Karen T Kotz   Date: 3/3/96
Karen T. Kotz. Laboratory Director

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1. Method Description

1.1.1. The purpose of this procedure is to provide instructions for planning, performing and reporting QA/QC audits within the laboratory.

1.1.2. This method is restricted to use by, or under the supervision of personnel experienced in the technique described.

1.2. Discussion

An Audit of the facility is performed for the following reasons:

1.2.1 To determine that contractual and regulatory obligations are fulfilled.

1.2.2 To determine that procedures and standards are being followed, and to insure good laboratory practice. These audits will include, but are not limited to the refrigeration unit temperatures, logbooks, balance calibrations, data, and standards traceability.

1.2.3 To establish that quality assurance objectives are met, including holding times, use of approved analytical methods, and stated objectives for precision and accuracy.

1.2.4 To serve as a management tool to evaluate appropriateness of quality assurance policies.

1.2.5 To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

1.2.6 To determine that records are prepared and maintained as required.

1.3 Documentation and Frequency

Documentation required is specified in the text and the frequency shall be as required by the QA Manager, but at least one technical audit shall be performed annually for each department. This audit may take place in parts, with additional and more extensive audits being scheduled as deemed necessary.

1.4. Definition of Terms

1.4.3 This section defines terms and acronyms as they are used in this SOP.

1.4.4 SOP: Standard Operating Procedure

1.4.5 QA: Quality Assurance

1.4.6 QC: Quality Control

1.4.7 Audit: A planned and documented activity performed to determine by investigation. examination. or evaluation of objective evidence the adequacy of and compliance with established procedure. instruction. and other applicable documents and the effectiveness of implementation. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

1.4.8 Auditor: Any individual: who performs or assists in the performance of any part of an audit. including technical specialists.

1.4.9 Lead Auditor: An individual who is qualified to organize and direct an audit. report audit findings. and evaluate proposed corrective actions.

1.4.10 Finding: Departure from approved procedures. program requirements. or other applicable documents that have. or in the immediate future could reasonably be expected to have, an adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **critical** discrepancy in the audit report.

1.4.11 Deficiency: Departure from approved procedures. program requirements. other applicable documents. or good management practices that. if not corrected in a timely manner. could reasonably be expected to have a future adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **minor** discrepancy in the audit report.

1.4.12 Discrepancy: Departure from approved procedures. program requirement, or other applicable documents that have. or may have an adverse effect on the adequacy or effective implementation of the Laucks QA program. This includes findings and deficiencies found during the course of an audit.

1.4.13 Recommendation: An observation or advise given to enhance current practices by any individual or department of the Laucks QA program. This would be ranked as a **recommended** item in the audit report.

2. Responsibilities

2.1 It is the responsibility of QA personnel, the auditor and audit leader to perform an audit according to this SOP and complete all documentation required for review.

2.1.1 QA Manager is responsible for the following:

- Approving each detailed audit plan
- Concurring with the adequacy of each audit report
- Issuing the audit report
- Tracking audit status through final closeout

2.1.2 If an audit team is used, the following responsibilities fall upon the Audit Team Leader. If an audit team is not used, the following responsibilities fall to the QA Manager:

- Developing the detailed audit plan
- Conducting pre-audit and post-audit conferences
- Supervising the conduct of the audit
- Preparing and signing the audit report

2.1.3 Management of audited departments is responsible for the following:

- Providing reasonable and timely access to personnel, facilities, and records, as required to support the audit process
- Providing timely and adequate response to audit reports, including determination and implementation of co- ective actions, as required.
- Verifying initial implementation of corrective action for deficiencies in their areas, if applicable.

2.2 Audits and reports are to be performed by personnel in the laboratory who have demonstrated the ability to evaluate processes in the laboratory with emphasis on Quality Control and Quality Assurance.

2.3 Final review and sign-off of each Audit Finding Report may be performed by either the QA Manager. Lab Director or department supervisor or designee.

3. Safety precautions

3.1. Safety Precautions

3.1.1. Auditors must adhere to the general laboratory health and safety policies during the course of the audit.

3.1.2 Protective eyewear must be worn in all applicable locations at all times during the course of the audit.

4. Calibration and Quality Control

Not applicable.

5. Operation procedures

5.1 General

5.1.1 Audit personnel may be selected and assigned audit responsibilities commensurate with their training and expertise and the special nature of the activities to be audited.

5.1.2 Audit personnel are independent of any direct responsibility for performance of any activity which they will audit. Persons having direct responsibility for performance of the activities are not involved in the selection of an audit team.

5.1.3 Audit team members shall have received appropriate indoctrination and training for auditing.

5.2 Audit Planning

5.2.1 The QA Manager. or designee shall develop an audit plan which shall be the basis for the audit. The audit plan is documented on Audit Plan Form (Se Appendix I).

5.2.2 The QA Manager shall develop an audit checklist appropriate to the activity or area be audited. The checklist should contain auditable requirements extracted from the QA Man applicable SOP's or guidance documents. such as EPA SW846. Checklists are designed for each Department by the QA Manager and can be accessed by the QA Department.

5.2.3 The QA Manager shall ensure that the checklist provides an adequate means for indicating whether the question is satisfactorily answered.

5.2.4 Audits are scheduled in a manner to provide coverage and coordination with ongoing QA program activities.

5.2.5 Audits are scheduled at a frequency commensurate with the status and importance of the activity. Within the audit program. each department of the laboratory and each element of the Laucks-QA program is audited. at a minimum. at least once annually.

5.2.6 The QA Manager notifies the audited department. in writing, prior to the audit to provide the subject and scope of the audit. audit schedule. and audit team members. if applicable.

5.3 Audit Performance

5.3.1 The QA Manager and (when required) the appointed audit team members shall proceed through the audit checklist recording evidence of compliance. discrepancies. or recommendations.

5.3.2 During the audit. the QA Manager or appointed team member shall use their best judgment to determine if there is a need to audit at a greater depth than the checklist indicates. If this is the case. the checklist shall be modified accordingly.

5.3.4 Objective evidence is examined. and essential information is recorded, such as the identification of specific evidence examined. specific details of discrepancies or adverse conditions. and applicable references.

5.3.5 The QA Manager shall identify each finding. deficiency. or recommendation in a QA audit report. Findings. deficiencies and recommendations will be listed by department and sequentially numbered in the QA audit report.

5.4 Audit Report

5.4.1 The QA Manager or his designee shall prepare an audit report which should address the following:

5.4.1.1 Date and location (Laucks-department) of the audit.

5.4.1.2 Purpose and scope of the audit.

5.4.1.3 Audit team members (when applicable) and the people contacted during the audit.

5.4.1.4 Description of items. including the rank. type and detail of the audit finding requiring corrective action. The description of the items must be in sufficient detail to enable investigation. evaluation. and correction of the finding. (See Appendix II - Audit Finding Report Form) The report may also include the area affected (See Table in Appendix III) and Finding Type (See Table in Appendix IV)

5.4.1.5 Due date for completion of corrective action plans.

5.4.2 The QA Manager shall issue the audit report to the appropriate levels of Laucks management within four following the audit. This report shall include a copy of each finding. deficiency and/or recommendation.

5.5 Audit Closure and Follow-Up

5.5.1 The appropriate Laucks Management (departmental supervisors, laboratory director hall investigate the reported finding. deficiency or recommendation and do the following:

5.5.1.1 Determine the actions required to correct the discrepancy.

5.5.1.2 Evaluate each discrepancy to determine the root cause of the problem and any generic implications.

5.5.1.3 Determine the corrective action required to correct the discrepancy and to prevent recurrence.

5.5.1.4 Document corrective action and indicate corrective action commitment date.

5.5.1.5 Sign. date. and return the completed form to the QA Manager within the assigned time frame given in the audit report.

5.5.2 The QA Manager shall evaluate each discrepancy/recommendation response. Inadequate or indeterminate responses shall be returned for reexamination of the problem and revised corrective action.

5.5.3 The QA Manager shall verify the corrective action, as stated in the response, and make sure it has been implemented and accomplished as scheduled.

5.5.4 An interim status report of corrective action completion may be issued

5.5.5 After verification of corrective action. the QA Manager shall issue a report stating that all corrective action has been completed and the audit is closed.

5.5.6 If a stalemate is reached concerning either the validity or resolution of an audit finding, affected personnel escalate the concern to the appropriate level of management to effect a resolution.

5.6 Records

The QA Manager shall ensure that the following audit documentation is maintained on file:

5.6.1 Completed audit checklist.

5.6.2 Audit Report (includes findings. deficiencies and recommendations).

5.6.3 Corrective Action (response to discrepancies).

5.6.4 Records pertaining to the completion of corrective action.

5.7 Audit Discrepancy Tracking

5.7.1 Audit discrepancies will be categorized to facilitate tracking and trending of recurrent problems. The categories are as follows:

- Logbook Maintenance
- Document Control Procedures
- QC Procedures
- Standard Operating/Quality Assurance Procedure
- Analytical Method
- Purchasing/Procurement Document Control
- Standards Preparation/Documentation
- Safety/Reagent Labeling or Storage

- Training/Records
- Good Laboratory Practices
- Other

5.7.2 Explanations of Categories Listed Above

5.7.2.1 Logbook maintenance findings include but are not limited to the following: logbooks not being maintained in accordance with Laucks policy. improper entries into logbooks. improper error corrections. logbooks not being kept up to date.

5.7.2.2 Document Control Procedure findings include but are not limited to the following: documents being maintained in such a way that is non-complaint with Laucks document control procedures (this includes archives. SOPs. QAPs. Chemical Hygiene Plan. HTVRs. and forms). records being stored in work areas for longer than 6 months. improper handling of controlled procedures.

5.7.2.3 QC procedure finding include but are not limited to the following: temperatures of ove and refrigeration units not being monitored in accordance with procedures, balances and pipettes not being verified as required.

5.7.2.4 Standard Operating Procedure and Quality Assurance procedure findings include any case where a procedure has not been followed in full and has not been documented on the applicable corrective action from.

5.7.2.5 Analytical methods findings involve cases where the approved and required analytical method has not been followed to the full extent and there is no documentation that communicates this.

5.7.2.6 Purchasing and procurement document control findings involve instances where the appropriate procedures have not been followed in full. This type of finding includes but is not limited to the following: un-approved use of standards or solvents. lack of certification documentation. etc.

5.7.2.7 Findings for standards preparation and standards documentation include but are not limited to the following circumstances: improperly prepared standards. improperly documented standard preparation. inadequate verification documentation, lack of documentation when procedures are not followed in full.

5.7.2.8 Safety and reagent/chemical labeling findings involve any deviation from approved safety and waste procedures and the chemical hygiene plan.

5.7.2.9 Training and training records findings involve lack of training records. and personnel performing analysis without appropriate qualification documentation.

5.7.2.10 Good Laboratory Practice findings involve significant figures. temperature monitoring. calibration techniques and other associated activities involved with safe and accurate laboratory practices.

6.1 References

Laucks Quality Assurance Plan

Applicable SOPs

Audit Database Tables

Appendix I

Audit Plan Form

LAUCKS Testing Laboratories

Audit Plan

Area to be Audited: ______________________________

Lead Auditor: ______________________________

Audit Team Members (if applicable): ______________________________
____

Date of Audit: ______________________________

Type of Audit: ______________________________

Checklist(s) to be Used: ______________________________
____
____
____

Individuals Contacted During the Audit: ______________________________
____
____
____

Audit Debrief Date: ______________________________

Report Issued Date: ______________________________

Signature of Lead Auditor: ______________________________

Signature(s) of Team Members: ______________________________
____
____

Appendix II

Audit Finding Report Form

Audit Finding Report

Audit Number: Example	Finding Number 1
Facility:	Audit Date:
Auditing Body:	Audit Type:
Lead Auditor:	Affected Area: GC-Semivolatiles

Related Findings:

Finding Rank: Minor   Repeat Finding?: No

Finding:

Corrective Action Response:

Opened By:	Date Opened:
Response By:	Response Date:
Corrective Action By:	Scheduled Completion Date:
Verified By:	Date Verified:

Depart	Department	DepartmentDescription	Sup ID #
ARCH	Archive	Archive of Documents in QA	0006
BP	Bottle Prep	Bottle Prep	0008
DM	Data Management	Data Management and Administrati	0008
EXT	Extractions	Extractions	0027
GCEF	GC-Extractable Fuels	Extractable Fuels by GC/FID	0038
GCS	GC-Semivolatiles	GC-Semivolatiles	0048
GCV	GC-Volatiles	GC-Volatiles	0038
MSS	GC/MS-Semivolatiles	GCMS-Semivolatile	0048
MSB	GC/MS-Semivolatiles & Volatile	GC/MS-Semivolatiles and Volatiles	
MSV	GC/MS-Volatiles	GCMS-Volatile	0038
SAF	Health and Safety	Health and Safety	0006
HPL	HPLC	HPLC	0038
IN	Inorganics	Metals and Wet Chemistry Office	0053
MIS	LIMS and MIS	LIMs and MIS	0070
MET	Metals	Metals and Metals Prep	0067
MTI	Metals Instrumentation	Metals Instrumentation	0067
MTP	Metals Preparation	Metals Preparation	0067
PM	Project Management	Project Management	0008
QA	Quality Assurance	Quality Assurance	0006
SM	Sales and Marketing	Sales Department	
SC	Sample Control	Sample Control	0008
SP	Special Chemistry	Special Chemistry	0053
WC	Wet Chemistry	Wet Chemistry	0053
YAK	Yakima Office	Yakima Office	0072

Appendix IV

Finding Type

ID of Finding Type	Finding Type
BA1	Balance - Not Certified Annually
BA2	Balance - Not Checked Daily With Class S Weights or as used
BA3	Balance - Weights Not Certified Annually
BA4	Balances - Weights used for calibration do not correspond to weights used for analysis
CA1	Corrective Action - Procedures Not Developed
CA2	Corrective Action - NVC Not Being Tracked
DL1	Documentation/Logbooks - Error and Corrections not be documented correctly
DL2	Documentation/Logbooks - incomplete columns, not properly bound
DL3	Documentation/Logbooks - Not Maintained or used
DL4	Documentation/Logbooks - Inadequate Review
DR1	Data Review - Not Being Performed
DR2	Data Review - Not Being Documented
DR3	Data Review - No SOP
DR4	Data Review - No QC Decision Matrix Available
EC1	Electronic Backup - Not Being Performed
EC2	Electronic Backup - Not inventoried For Retrieval
GL1	Good lab practice - misc GLPitems
MD1	Methods - No SOP/cribsheet available at time of audit
MD2	Methods - SOP/Cribsheet in use not current controlled version
MD3	Method- controlled SOP/Cribsheet is not being followed or doesn't match current practice
MD4	Methods - The controlled SOP is Non-compliant with the referenced published method
MD5	Methods - SOP/Crib sheets in use & not controlled, meaning draft or handwritten SOPs in use
PE1	Performance Evaluation Samples - Results are outside warning limits, check for error
PE2	Performance Evaluation Samples - Results are outside control limits, not acceptable
PE3	Performance Evaluation Samples - Results included misidentified compounds, not acceptable
QA1	QA - QAP/SOP Document Control Not in Place or Used
QA2	QA - Precision and Accuracy Data Not Current
QA3	QA - MDL.IDL Not Current
QA4	QA - QC Limits Not Determined or Maintained
QA5	QA - Control Charts Not Developed or Maintained
QP1	QAPlan - No QAP Available
QP2	QAPlan - Outdated And Needs Revision
QP3	QAPlan - Has Major Discrepancies With SOPs or practices of the day
RC1	Records Control - Logbooks Not Controlled
RC2	Records Control - Filing not maintained per SOP
RC3	Records Control - No SOPs to desribe System
RC4	Records Control - Not mentioned in QAP
RC5	Records Control - Archiving inadequate
SC1	Sample Control - Building not secured
SC2	Sample Control - COC not establisned or maintained per client requirements
SC3	Sample Control - Temp/pH not monitored for all regulatory samples
SF1	Safety - No SOP
SF2	Safety - Not Adhering to SOP or Chemical Hygiene Plan
SF3	Safety - Not Adhering to Good Lab Safety Practices
ST1	Stds/Reagents - No SOPs for preparation
ST2	Stds/Rgnts - Prep record inadeq./or not traceable
ST3	Stds/Reagents - Expiration Date Misused
ST4	Stds/Reagents - Not Labeled Properly in the laboratory
SW1	Software - Not Verified and Documented
TH1	Thermometer - NIST Not Available
TH2	Thermometer - NIST Not Evaluated Annually
TH3	Thermometers - Not Calibrated Annually
TH4	Thermometers - Correction Factor Not Applied or misapplied
TH5	Thermometers - Temp. Not Recorded Daily or As Used
TR1	Training - No Formal Program or Documentation
TR2	Training - Incomplete Forms (eg Proficiency, Hrs)
TR3	Training - Not Maintained Consistently

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1018

Title: **Overview of Review and Approval Practices for Validatable Data Packages**

Revision history:

Number	Date
0	06/17/96

Written by: Harry Romberg Date: 6-17-96
Harry Romberg, Quality Assurance Officer

Approved by: Karen J Kotz Date: 6/20/96
Karen Kotz, Laboratory Director

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1 Description

1.1.1 This SOP is intended to provide an overview and general organization of data review practices employed for validatable packages. The actual data review processes and check lists specific to those types of analyses are covered in specific SOPs. A schematic diagram of the general review process is provided in Appendix I.

1.1.2 Validatable packages are often similar to the Contract Laboratory Program (CLP) presentation, although the actual analyses themselves and the applicable quality control (QC) may be from SW 846 or other references. If such is the case, the CLP format would be modified to meet the requirements of the referenced methodology. However, the overall review process remains the same.

1.1.3 In-house (non-validatable) data packages receive much of the same review but do not necessarily follow the same process or the same level of documentation. It is not the intent of this SOP to outline the process for these data.

1.1.4 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described function.

2. Equipment List

2.1 Equipment

2.1.1 Data package or the portion of the data package to be validated

2.1.2 SOPs, including associated checklists, for the validation of the analyses of interest

2.1.3 Access to computer programs, etc. which may be required in order to complete the review process

3. Responsibilities

3.1 Analyst

3.1.1 It is the responsibility of the analyst to provide the first level of data review and to ensure that all criteria have been met or their failure addressed prior to releasing the data for the next level of review. The analyst may only be the first level of review but is the most important in ensuring that the reported values reflect what was actually present in the samples. It is particularly important that the analyst be proactive in determining any actions that need to be taken in order that they may be completed within the holding time for that analysis and within the turnaround time required by the client.

- The analyst must ensure that the instrument was functioning properly at the time of analysis
- The analyst will ensure that all data comply with the method and project-specific requirements and that any deviations or failures to meet criteria are documented in the project file.
- The analyst must check to see that all calibration criteria were met
- The analyst must review all quality control data and ensure that criteria were either met or corrective action taken. This action may vary anywhere from simple narration in the report to re-analysis of the sample set, depending upon the QC failure and the method requirements.
- The analyst will review the final data to see that they make sense, that is, the values determined are reasonable, do not disagree with other information the analyst may be aware of, and that the calculated values appear to agree with the raw data.
- The analyst will either transcribe the data into the LIMS or will pass data to the person responsible for transcription in a format which can be easily interpreted.

3.2 Peer or Secondary Review

3.2.1 Data must receive a second level of review from a peer analyst. This analyst should be a person who is familiar with and capable of performing the analysis themselves. If there is no peer analyst available because the analyst in question is the only one experienced with the analysis or for other critical reasons, another qualified individual may substitute for the peer analyst. This person must still be familiar with all aspects of the calculations being performed and the relationships between data and performance of the method in order that the review can be properly conducted. The peer analyst reviewer must:

- Check 100% of the manual entries for transcription errors
- Check 100% of manual calculations for accuracy
- Spot check at least 10% of computer calculations to verify program validity
- Check for completeness of raw data or supporting materials
- Confirm spectral assignments and identification of TICs

- Check for appropriate use of significant figures and rounding
- Check reported values for dilutions
- Check for compliance with Method and project-specific requirements
- Check for reasonableness of data

3.3 Supervisor

3.3.1 The responsible supervisor or a designated alternate for the area in which the analysis is conducted must provide a technical review of the reported data. This level of review need not be as detailed as the peer review but must include:

- Checking for reasonableness and sensibility of the reported data
- Checking for completeness of the reported information
- Checking for compliance required QC practices including those specified in the Method and those that are project-specific.
- Checking for descriptions of deviations from Method and project-specific QC requirements
- Checking the information in the report narrative for sensibility

3.4 QA Review

3.4.1 QA cursorily reviews most data and periodically, in conducting data audits, reviews select packages more thoroughly. The cursory reviews are generally performed just prior to release of the data. In depth reviews almost always occur after release of the data and are intended more for a review and assessment of laboratory data and processes rather than an assurance of performance on that particular data package. Should quality issues arise that have a critical negative impact on the package being thoroughly reviewed, however, QA may call for more specific corrective action. QA may choose to go into any depth in review of data packages, but in general, most reviews will consist of:

- Checking for compliance with required QC practices
- Checking for reasonableness and sensibility of reported data
- Checking for deviations from Method or project QC requirements
- Checking for compliance with SOPs (periodically)

3.5 Project Manager

3.5.1 Project managers do not perform technical reviews but do review case narratives to ensure compliance with contractual agreements. Their responsibilities include:

- Reviewing to ensure that the client requested methodology was used and referenced
- Ensuring that sample entry comments were incorporated, and that concerns that were raised during the course of analysis which required client communication and decisions have been incorporated.
- Reviewing and signing project narratives.
- Reviewing the billing to ensure that the proper invoicing has occurred in conjunction with contractual agreement.

3.6 Management

3.6.1 Senior management reviews case narratives and other components of data packages, should they find cause. They are the parties responsible for approving the release (signing) of reports.

4. Operation Procedures

4.1 General

4.1.1 The processes described below are general. Specific QC and practices, including most of the corrective actions resulting from QC failures are generally described in the appropriate SOPs. The specifics of the review process for individual analyses are specified in the respective data review SOPs along with their associated checklists.

4.1.2 The duties of individuals responsible for various levels of review are specified in the Responsibilities section of this SOP. It is the responsibility of each reviewer to be familiar with this SOP and those specific to their function.

4.2 Analyst

4.2.1 The analyst must be cognizant of the entire analytical process and document anything out-of-the-ordinary that goes on during the analysis. This may include on-the-spot corrective action, such as dilution and re-analysis. The analyst must also review the data during the production of final results to ensure that all criteria are met and that all appropriate commentary regarding the analysis and any extraordinary steps are clearly noted.

4.2.2 The analyst will then assemble the final data package according to the SOP and submit the data for review to the secondary reviewer. The work of the analyst is the most critical in the review process as this ensures the timely processing of the samples in order to meet holding and turnaround times.

4.2.3 When completed with the data package, the analyst will pass all of the associated materials along to the second reviewer.

4.3 Peer or Secondary Review

4.3.1 The secondary review will usually include use of the checklists associated with the data review SOPs. If in doubt, the secondary reviewer will ask the analyst for further information and not just pass along problems to the next level. In consultation with the supervisor or QA, data may be returned to the analyst for corrective action.

4.3.2 The secondary reviewer will pass the data and checklist along to the supervisor

4.4 Supervisor

4.4.1 The area supervisor or designate will perform the functions outlined under the Responsibilities section, paying special attention to data review checklist items which do not meet method specifications. The supervisor may determine that corrective actions are necessary in the pursuit of data of adequate quality or may consult with QA where the optimal practice is questionable. The supervisor should ensure that corrective actions are all completed and all report commentary is sound prior to submitting the data to the reports department.

4.5 Reporting

4.5.1 The reporting group assembles the respective data packages but bears no responsibility for review other than to ensure that all of the analyses are present in the package, that everyone has input their respective commentary into the report narrative, that all narrative comments have been printed and the appropriate parts of the data package have been assembled. This aspect is detailed in an SOP designed for that purpose.

4.6 Quality Assurance

4.6.1 QA performs cursory reviews of most narrative and data packages before release. QA may call for corrective action at any level should problems be observed which have not been dealt with in an appropriate manner prior to this late stage of reporting. Responsibility to spot and have errors corrected, however, must not be left up to QA if they are spotted earlier or the analysis and reporting of results will almost certainly be delayed.

4.6.2 QA will also perform a more thorough review of select data packages, the scope of which is at the discretion of QA and is not addressed in this SOP. Such review will be more detailed, however, and corrective actions may result which will impact the immediate data or, more likely, affect the processes involved in collecting, reviewing, or reporting data in general.

4.7 Project Management

4.7.1 Project managers review and sign project narratives. They will review only to ensure that the client requested methodology was used and referenced, that sample entry comments were incorporated, and that concerns that were raised during the course of analysis which required client communication and decisions have been incorporated. They must also review the billing to ensure that the proper invoicing has occurred in conjunction with contractual agreement. They may perform these tasks either before or after QA review.

4.8 Management

4.8.1 Management will review and release (sign) narratives.

5. Reports

5.1 Data Review and Signatures

5.1.1 Data review forms are provided in individual data review SOPs.

5.1.2 Analyst/reviewer signatures occur on organics cover pages. Inorganics review signatures occur on data cover pages and supervisor signatures are included on both metals and conventional chemistry packages.

5.1.3 Management signatures appear on all final reports.

6. References

Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center. February 1996

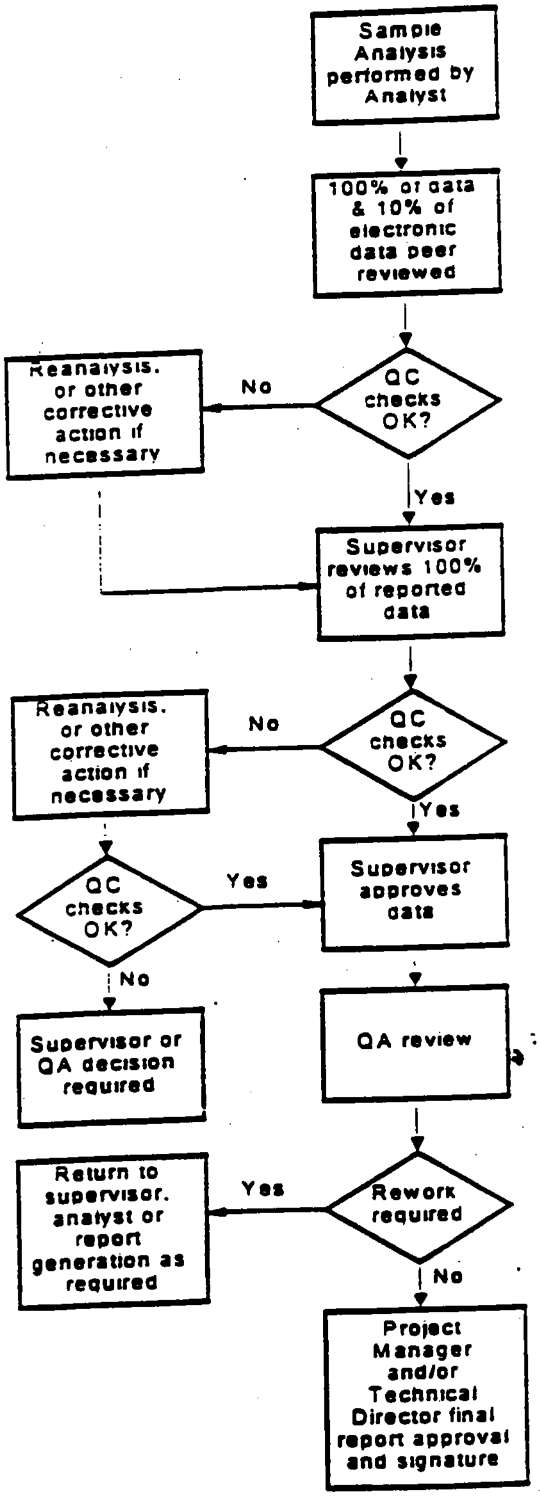
Laucks SOPs

LTL-7001	Metals Data Review
LTL-7002	EPA Inorganics Data Review
LTL-8001	GC Hydrocarbons Data Review
LTL-8002	GC Pesticides/PCBs Data Review
LTL-8003	GC Herbicides Data Review
LTL-8004	GC Volatiles Data Review
LTL-8005	GC Gas/BTEX Data Review
LTL-8201	GC/MS Volatiles Data Review
LTL-8202	GC/MS Semivolatiles Data Review
LTL-8301	HPLC Aromatics 8310 Data Review
LTL-8302	HPLC Ordnance 8330 Data Review
LTL-9001	Inorganic Conventionals Data Review

Appendix I

Data Review Schematic

Data Review and Approval



- -100 % manual entries for transcription errors
- -100% of manual calculations for accuracy
- -10% spot check of computer calculations
- -Check for completeness of raw data or supporting materials
- -Confirm spectral assignments and identification of TICs
- -Check for appropriate use of significant figures and rounding
- -Check for compliance with Method or Project QC requirements
- -Check for descriptions of deviations from Method or Project QC requirements
- -Check reported values for dilutions
- -Check reasonableness of data

- -Check for reasonableness of reported data
- -Check for completeness of the reported information
- -Check for compliance with required QC practices
- -Check for descriptions of deviations from Method or Project QC requirements
- -Check the information for the report narrative

- -Check for compliance with required QC practices
- -Check for reasonableness of reported data
- -Check for descriptions of deviations from Method or Project QC requirements
- -Check for compliance with approved SOPs (periodically)

P:\SOP\QA_SOPS\RVW_CHT1.GFC 6/13/96

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1019

Title: **Controlling, Maintaining, and Monitoring Laboratory Logbooks**

Revision history:

Number	Date
0	4/11/96
1	5/13/98

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 5-13-98

Approved by: Kathy E Kreps
Kathy Kreps, Laboratory Director
Date: 5-18-98

Controlled Document

No. 20 **Assigned to:** Tetra

Table of Contents

1. Introduction and Scope

1.1 Scope

1.1.1 The maintenance of logbooks is essential to monitoring all aspects of laboratory operations including instrument and method performance and in tracking analyses. It is also important to confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. This document applies to all personnel involved in the preparation, control and use of laboratory notebooks.

1.1.2 More specific instructions for maintaining logbooks can be found in pertinent SOPs, such as LTL-1007 "Maintaining Instrument Records and Logbooks" or LTL-1005 "Analytical Balances" or others specific to other laboratory operations.

1.2 Purpose

1.2.1 The purpose of this SOP is to define the practices used to maintain control and use of laboratory logbooks. This SOP is not intended as a specific description of any particular logbook type but covers the practices that must be in place for all logbooks employed at Laucks.

1.3 Definition of Terms

1.3.1 Logbook - Any bound or unbound document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs, standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs, or any other regularly maintained record of activity.

2. Requirements

2.1 Control

2.1.1 Documents that specify quality requirements or activities affecting quality or evidentiary activities shall be controlled to ensure that correct documents are being used and properly archived when completed.

2.2 Maintenance

2.2.1 Quality assurance records (logbooks) shall be compiled and maintained in accordance with approved procedures.

2.3 Monitoring

2.3.1 Logbooks should be periodically monitored to ensure they are being properly maintained and information is being correctly recorded. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring.

3. Responsibilities

3.1 Quality Assurance Manager

- Maintain the logs for control of laboratory notebooks and provide control numbers and labels as required.
- Approve format and proposed content of laboratory notebooks; minor changes to pre-printed forms do not need QA approval as long as their basic content does not change.
- Maintain master copies of notebook pages (in instances where pre-printed pages with a specific format are used); this may be in electronic or hardcopy form or both.
- Monitor satisfactory implementation of the requirements of this SOP

3.2 Responsible Supervisor

- Determine the format and content of notebooks used in their respective areas.
- Ensure that QA has been provided with an electronic version of all pre-printed logbook pages in order that they are later available for reprinting or editing.
- Ensure that all laboratory notebooks are properly labeled, including the appropriate control number.
- Ensure that personnel are adequately trained in the proper use of laboratory notebooks
- Periodically review laboratory notebooks to verify satisfactory implementation of the requirements of this SOP. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

3.3 Analyst

- Ensure that they are using the appropriate logbook and understand how to properly fill in the required fields.
- Ensure that any new logbook has been given a logbook number by QA before beginning to use it.
- Ensure that the logbook is clearly identified with an instrument ID and purpose or other appropriate title which will enable the analyst to easily identify the logbook.
- Ensure that if pre-printed logbook pages need to be modified, the modifications are approved by their supervisor and that an electronic copy or, if requested an original hardcopy have been provided to QA.

4. Procedure

4.1 Notebook Structure

4.1.1 Laboratory notebooks may be either bound or unbound as described below. Most logbooks should be bound in some fashion but it is recognized that this is not always possible, such as for vendor service records. These records may be stored in 3-ring binders or other suitable notebooks.

4.1.2 In some instances, logbooks may be created from instrument printouts or other pages that do not lend themselves to being pre-bound. In these instances. the log sheets may be stored in a 3-ring binder or other storage until enough sheets have been accumulated to have them bound with the laboratory comb binder.

4.1.3 All logbooks whether bound or unbound must be controlled by QA as designated by the appropriate QA Book Number label (see example in Appendix 1).

4.1.4 Bound notebooks shall conform to the following:

- Where feasible, binding will be of a type that will make the removal and reinsertion of pages readily noticeable.
- If pre-printed and bound, all pages will be sequentially pre-numbered. If the format of the notebook permits the use of the reverse side of the pages, both sides of each page will contain a sequential page number.

- Each page of the pre-printed bound notebook will contain, as a minimum, the laboratory name, logbook title, and sequential page number. Other elements may also be necessary for any specific logbook.

4.1.5 Unbound notebooks shall conform to the following:

- Unbound pages will be contained in a binder or folder that provides protection from damage.
- Each unbound page will contain a unique identifier (e.g., run number/date). For identification purposes, a continuous printout on fanfold computer paper requires only one identifier unless the sheets are separated.
- As noted above in 4.1.2, some unbound logbooks may eventually be bound if practical.

4.1.6 All notebooks will contain the following information on the cover:

- Laboratory name, Laucks Testing Laboratories, Inc.
- Control number assigned by the Quality Assurance Officer
- The department to which the logbook was issued
- The use of the logbook (i.e. balance calibration. instrument run-log, etc.)
- The department book number or title uniquely identifying that book, as required to identify the specific use of the book. This may include an instrument number or other logbook ID (such as a standards logbook ID). This is in addition to the QA logbook number.
- Start Date, the date on which the first entry was made
- End date, the date on which the last entry was made

4.2 Control of Logbooks

4.2.1 The QA Officer will maintain a master log of laboratory notebooks that contains as a minimum, the following information:

- Unique control number for each logbook
- Logbook title, which should reflect the type of information to be entered.

- Department to whom issued, for accountability only. A logbook will generally be assigned to a work station or function, and in no way is a laboratory notebook to be considered a "personal" notebook.

- Date issued, for accountability only.

- Date closed, for accountability.

4.2.2 Master sheets for each logbook will be maintained by the QA Officer and will be utilized for producing notebooks when required.

4.3 Use of Laboratory Logbooks

4.3.1 The notebook is the basic document for recording information. Entries should be made into the notebook in real time, not written on scratch paper and transferred later.

4.3.2 Handwritten entries should be legible and entered in black or blue indelible ink.

4.3.3 Computer-generated data should be printed out and collected at appropriate times to represent the activities being recorded.

- Computer printouts may be either placed in unbound notebooks as described above, or inserted into bound notebooks.

- Computer printouts or other material inserted into bound notebooks must be securely fastened (tape is preferred) in such a way that removal and insertion of material can be determined readily.

4.3.4 When information from related activities is recorded in more than one notebook, provide adequate cross-reference information in all affected notebooks so that all pertinent data can be readily accessed.

4.3.5 Do not skip pages when entering data. For example, if data is not readily available for entry, do not leave space for later entry. Enter the data when it becomes available and provide adequate cross-references if required.

4.3.6 In cases where partial or complete pages must be left blank and not used, indicate the unused portion by placing a horizontal line at the beginning and end of the unused portion and connecting opposite ends of the horizontal lines with a diagonal, resulting in a Z-shaped figure. The individual striking out the blank area will initial and date the diagonal.

4.3.7 Errors or other changes must be deleted in a similar fashion or with a single-line cross-out which has been initialed and dated. **No erasures, overwriting, white-out or multiple-line cross-outs (blacking out) are acceptable.**

4.3.8 When pre-printed formats are used and all possible entries are not required, the remaining blanks may be struck out with a Z as described above, or entries such as N/A may be placed in the unused blanks.

4.3.9 The individual entering information into the notebook shall initial and date each page used, or in the case of logbooks with ongoing records which do not occupy the entire page, such as maintenance logs or balance logs, each individual entry.

4.4 Supervisory Monitoring of Laboratory Logbooks

4.4.1 Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

4.4.2 Logbooks should be reviewed using the review items provided in Appendix II, although it is not necessary to actually document the review using this checklist.

4.4.3 Errors should be formally brought to the attention of the responsible individual through the use of Corrective Action Forms. If errors are correctable or items can be corrected for legibility problems, they should be corrected using the proper error correction technique.

4.4.4 Logbooks that have been reviewed are marked with a fluorescent yellow or other colorful label that looks similar to the label in Appendix III.

4.5 QA Monitoring of Laboratory Logbooks

4.5.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained. This will generally be done approximately annually as part of routine audits. This observation does not preclude the requirement for supervisory review.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.: ____________________

Issued To: ____________________

Used For: ____________________

Dept. Book No.: ____________________

Start Date: ____________________

End Date: ____________________

Appendix II

Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced.

Appendix III

Example Logbook Review Label

Logbook pages ______through_______
have been reviewed for completeness and
spot-checked for accuracy>

Initials:______ Date:___________

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1020

Title: **The Integration of IC, GC, HPLC, and GC/MS Peaks**

Revision history:

Number	Date
0	08/26/96
1	08/21/97

Revised by: Monica Carr, Organics Division Manger — Date: 8/25/97

Reviewed by: Harry Romberg, QA Officer — Date: 8-25-97

Approved by: Karen Kotz, Laboratory Director — Date: 8/25/97

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the integration process for chromatographic data, the procedures for manual integration, and the procedures for documenting manual integration.

1.1.2 Integration identifies peaks found in the data collected during data acquisition and characterizes them. The software uses the integrated peaks to determine the identity and quantity of compounds in the samples. The peak area, peak height, peak type, baseline, and retention time of each peak in a chromatogram are determined by integration. Some peaks, due to limitations of the software, will need to be manually integrated. The manual integration process must be documented. This documentation will include a brief description of why it was necessary, who did it, when was it done, and a hardcopy of the re-integrated peak.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Definition of Terms

1.2.1 Manual Integration - any intervention by an analyst or supervisor to change the peak area, peak height, baseline, peak type, or retention time of a chromatographic peak.

2. Software

2.1.1 GC Acquisition-HP/LAS and EZChrom
2.1.2 GC/MS-Teknivent/EnviroQuant
2.1.3 GC-Target

3. Responsibilities

3.1 Analyst

3.1.1 The analyst is responsible for reading and understanding this SOP and that which is applicable to the method of analysis. The analyst must also perform and document all manual integrations as specified in this SOP.

3.2 Supervisor

3.2.1 The supervisor or designated data reviewer must verify that all manual integrations are performed according to this SOP. The supervisor or designated data reviewer must document that this verification has occurred per the applicable Data Review, Validation, and Reporting SOP.

4. Operation Procedures

4.1 Integrator operation

4.1.1 The ideal chromatogram has perfectly symmetric peaks separated from each other with periodic baseline points. It is common to encounter split peaks, deformed peaks, merged peaks, sloping baselines, noise, spikes, shoulders on peaks, and a host of other calamities.

4.1.2 To maximize the chances of obtaining ideal chromatograms: optimize the chromatography.

4.1.3 Peak recognition and integration sequence - As the integrator scans the data, it examines the slope (vertical distance between points) and curvature (positive or negative). So long as these remain within preset bounds data is interpreted as the baseline. If the bounds are exceeded, a peak may be starting. If the condition persists, the integrator decides that it is on the upslope of a peak.

4.1.4 The curvature changes to negative about halfway up the peak. This is the inflection point where the peak starts to round over approaching the apex. Passing the top, the slope becomes negative and the integrator is on the downslope. Another inflection point comes on the downslope and finally the peak returns to the baseline (Figure 1).

4.1.5 Finite width of integration slices - The slope changes from positive to negative at the top of the peak. However for area slices having finite width the integrator can only determine which slice contains the peak apex. To get better values for the retention time and peak height, the integrator takes the slice containing the apex and one slice on either side, fits them to a quadratic equation, and solves the equation to find the highest point (Figure 2).

4.1.6 Optimizing peak recognition - The best conditions for recognizing isolated symmetric peaks on a quiet baseline is to match the peak width parameter to the measured width of the peaks at half height. Threshold should be a few units less than the highest value still capable of detecting the peak. When peaks cluster together or the baseline slopes or is noisy, these ideal values must be modified. Figure 3 shows the effects of changing the values.

4.1.7 Manual integration - It is important that the analyst is familiar with the compounds that are routinely analyzed. Knowing the response and peak shape of the standard is important for consistency in integration. It is best to optimize the method to process data so that manual intervention is minimized and peak integration is more consistent. If manual integration is performed, it is important to be consistent for a given analyte in the standards, blanks, spikes, and samples.

4.1.8 Allowable manual integrations - Some common reasons for manual integration are:

4.1.8.1 Split peaks (attachment 1)

4.1.8.2 Tailing (attachment 2)

4.1.8.3 Retention time shifts (attachment 3).

4.1.8.4 Mis-identification (attachment 3)

4.1.8.5 Merged peaks (attachment 3)

4.1.8.6 Secondary ions or qualifier ions (attachment 4)

4.1.8.7 Baseline shifts (attachment 5)

4.1.8.8 Skimming versus dropped baseline (attachment 6)

4.1.9 Improper manual integrations - These practices are not allowed and warnings up to and including termination of employment will follow any documented cases of improper manual integration. If you are unsure about a manual integration ask your supervisor or QA.

4.1.9.1 Adding area by including other peaks. (attachment 7)

4.1.9.2 Improper baseline - this includes the practice of having the baseline moved up the side of a peak to decrease the area. (attachment 8)

4.1.9.3 Changing a proper integration to make the peak "in".

4.1.10 Special rules for fuel analyses (*e.g.*, gasoline, diesel) - The integration of these multi-component analytes requires special integration rules. The area of all peaks (with the exception of the surrogates) and the area of the non-resolved components (hump) are grouped together to determine the quantity of the analyte. The baseline is fixed at the start and held at a constant level for the entire run. When integration of the baseline for fuel analyses is performed, the quantitation report will not be dated and initialed by the analyst. Instead an explanation of the baseline integration will be documented on the quantitation report or in the appropriate SOP and discussed in the sample narrative. Some acceptable multi-analyte integrations are shown in attachment 9.

5. Documentation

5.1.1 When any manual integration is performed , a graphic copy of the peak with the integration marks is generated and put into the folder with the chromatograms and quantitation reports for that sample. The three different software systems used in the laboratory are listed below with the commands for generating the copy. On the quantitation report the analyst must initial, date, and give a brief description of the reason for the manual integration (table 1). The manual integration must also be documented in the associated sample narrative. The supervisor will look at each manual integration during data review and complete the summary on the data review or QC checklist.

5.1.2 Target: After changing the integration, exit and save in Target Review.

5.1.3 GC/MS Teknivent EnviroQuant: After using Qedit to change the peak click on 'Graphics Report to Printer'

5.1.4 EZChrom: After changing the integration, toggle the "Reanalyze" key and print.

5.1.5 LAS - After changing the integration, print the chromatogram.

Table 1

Manual Integration Key

M = Manual integration due to missed peak or irregular peak shape.
MS = Manual integration due to split peak.
MR = Manual integration due to retention time shift.
MI = Manual integration of correct isomer.
MT = Manual integration due to peak tailing.
MB = Manual integration due to irregular baseline.

6. REFERENCES

6.1.1 Target Manual, Thru-Put Systems, Inc., Target Compound Analysis Software User's Guide, 1994.

6.1.2 Envirolink User's Manual, TEKNIVENT, 1994

6.1.3 EZChrom Chromatography Data System, Scientific Software, Rev. 6.6, 1995.

6.1.4 Wisconsin DNR Newsletter, October 1994.

6.1.5 SOP# LTL-1018, Standard Operating Procedure for Review and Approval Practices for Validatable Packages.

6.1.6 SOP # LTL-8005, Standard Operating Procedure for GC Gas/BTEX Data Review.

6.1.7 SOP #LTL-8004, Standard Operating Procedure for GC Volatiles Data Review.

6.1.8 SOP #LTL-8001, Standard Operating Procedure for GC Hydrocarbons Data Review.

6.1.9 SOP #LTL-8301, Standard Operating Procedure for HPLC Aromatics 8310 Data Review.

6.1.10 SOP #LTL-8302, Standard Operating Procedure for HPLC Ordnance 8330 Data Review.

6.1.11 SOP #LTL-8201, Standard Operating Procedure for GC/MS VOA Data Review.

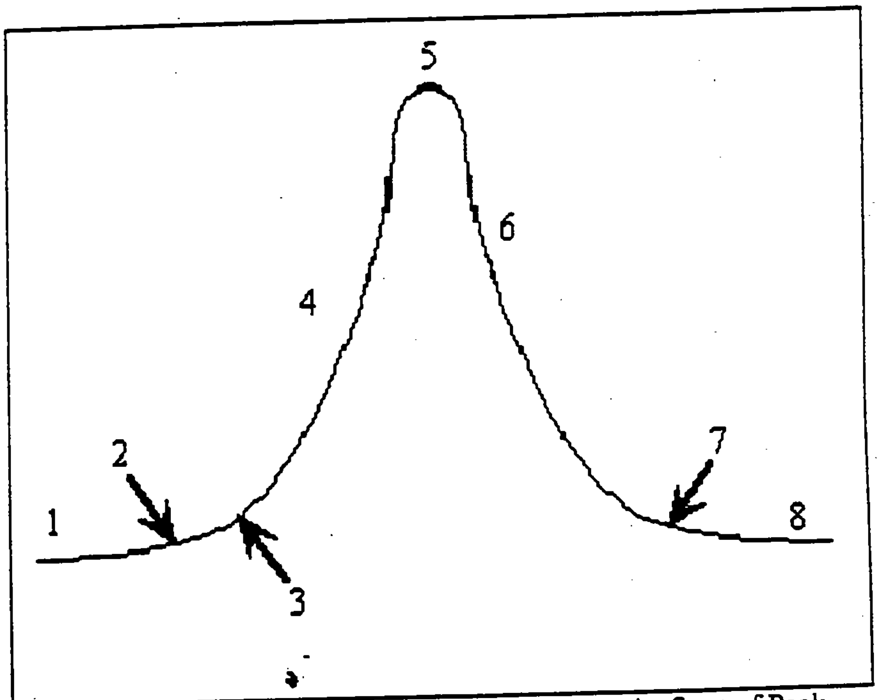
6.1.12 SOP #LTL-8202, Standard Operating Procedure for GC/MS Semivolatile Data Review.

Appendix I

Fig 1.

The sequence for finding a positive peak is:

1. Slope and curvature within limits	track baseline
2. Slope and curvature above limits	perhaps a peak?
3. Slope remains above limit	here's a peak!
4. Curvature becomes negative	front inflection point
5. Slope becomes negative	top of peak
6. Curvature becomes positive	rear inflection point
7. Slope and curvature within limits	approaching end of peak
8. Slope and curvature remain within limits	end peak, track baseline



Steps 3,5, and 8 define Cardinal Points, which are the Start of Peak, Apex, and End of Peak Respectively

Fig 2.

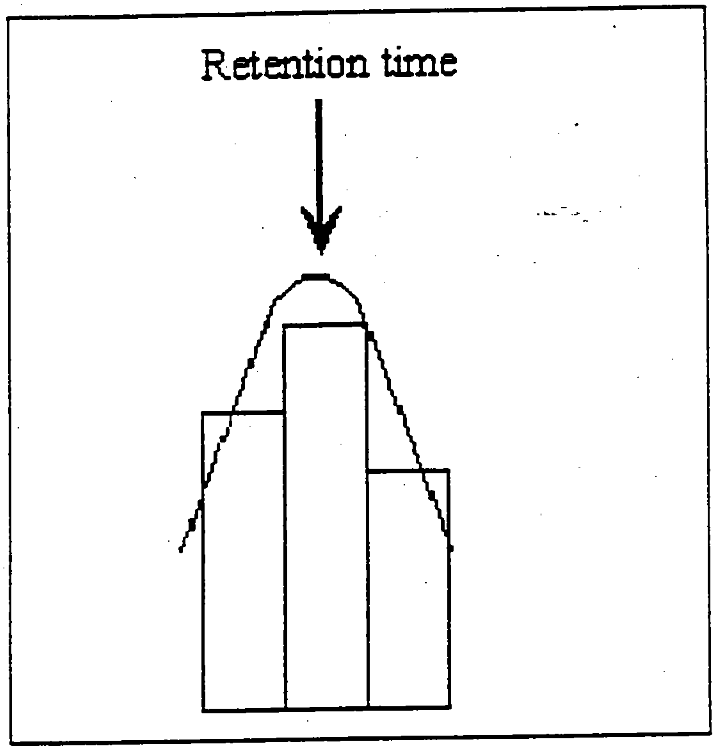
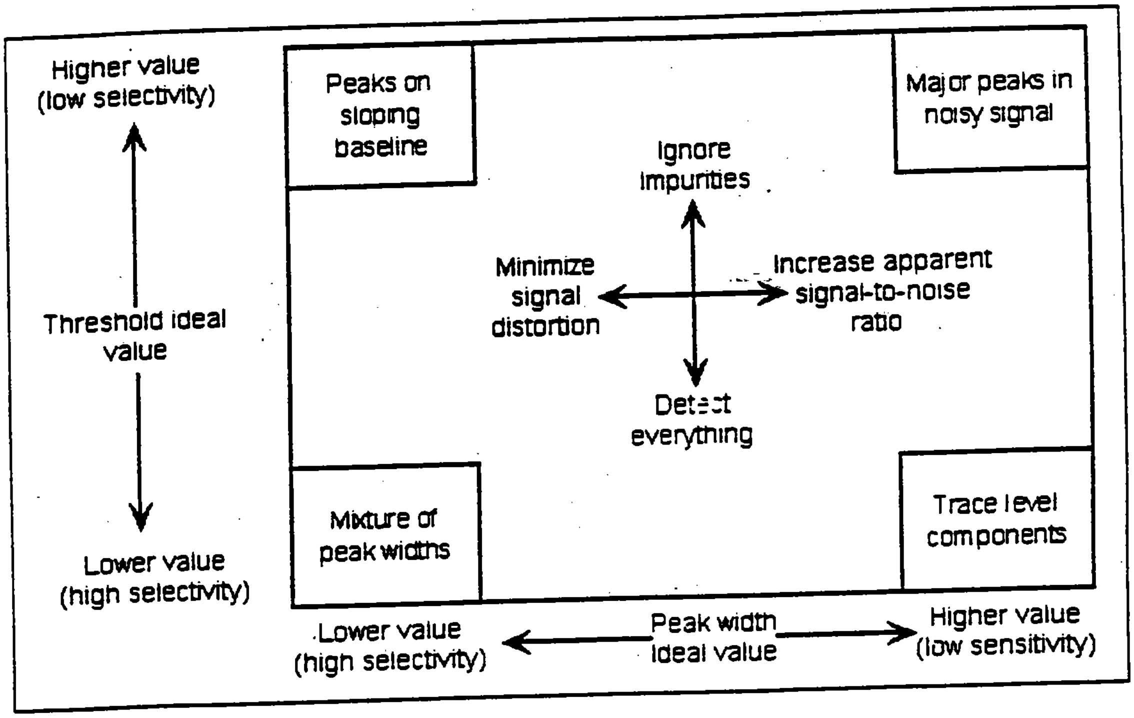
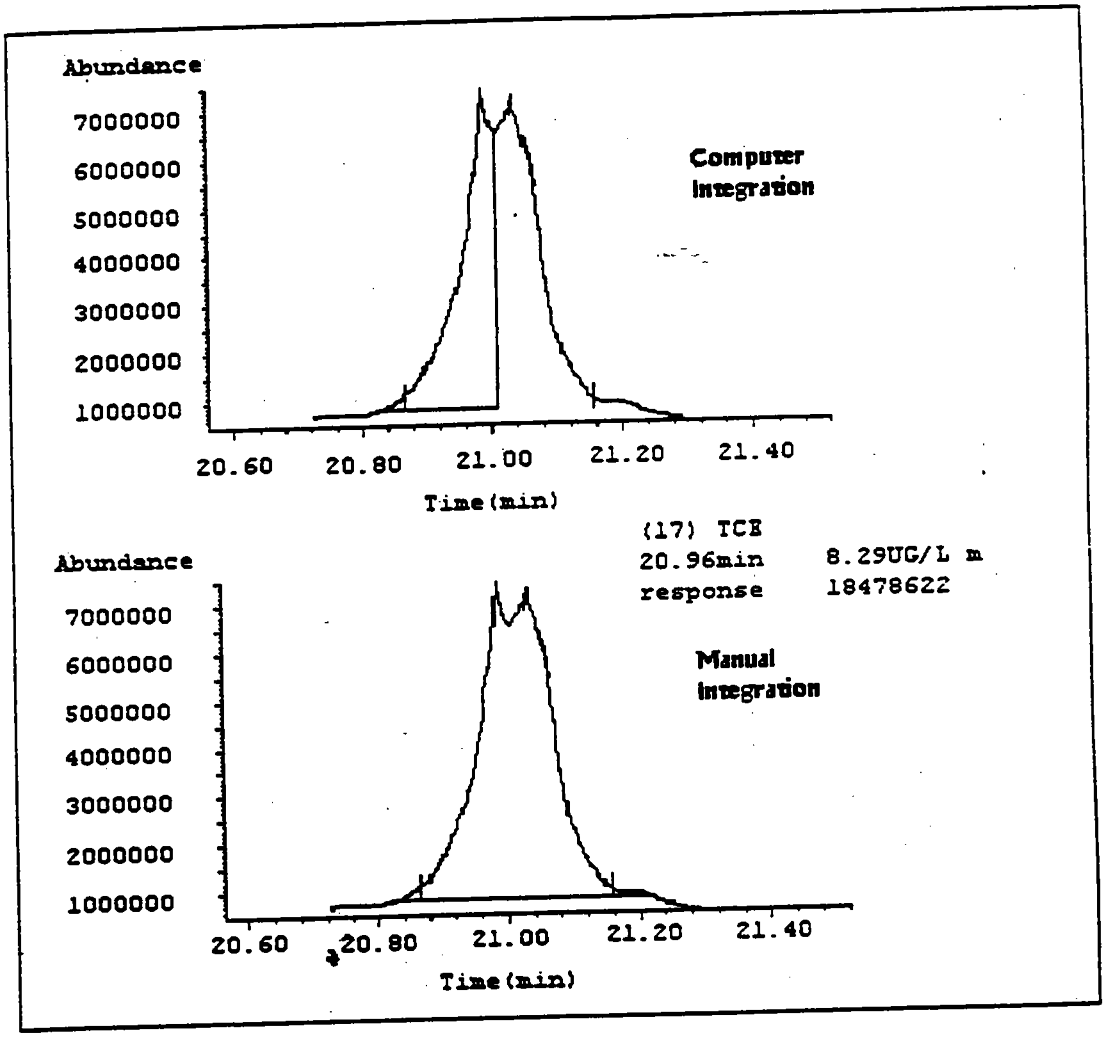


Fig 3.


Higher value
(low selectivity)
Threshold ideal
value
Lower value
(high selectivity)
Peaks on
sloping
baseline
Major peaks in
noisy signal
Ignore
impurities
Minimize
signal
distortion
Increase apparent
signal-to-noise
ratio
Detect
everything
Mixture of
peak widths
Trace level
components
Lower value
(high selectivity)
Peak width
ideal value
Higher value
(low sensitivity)

Appendix II

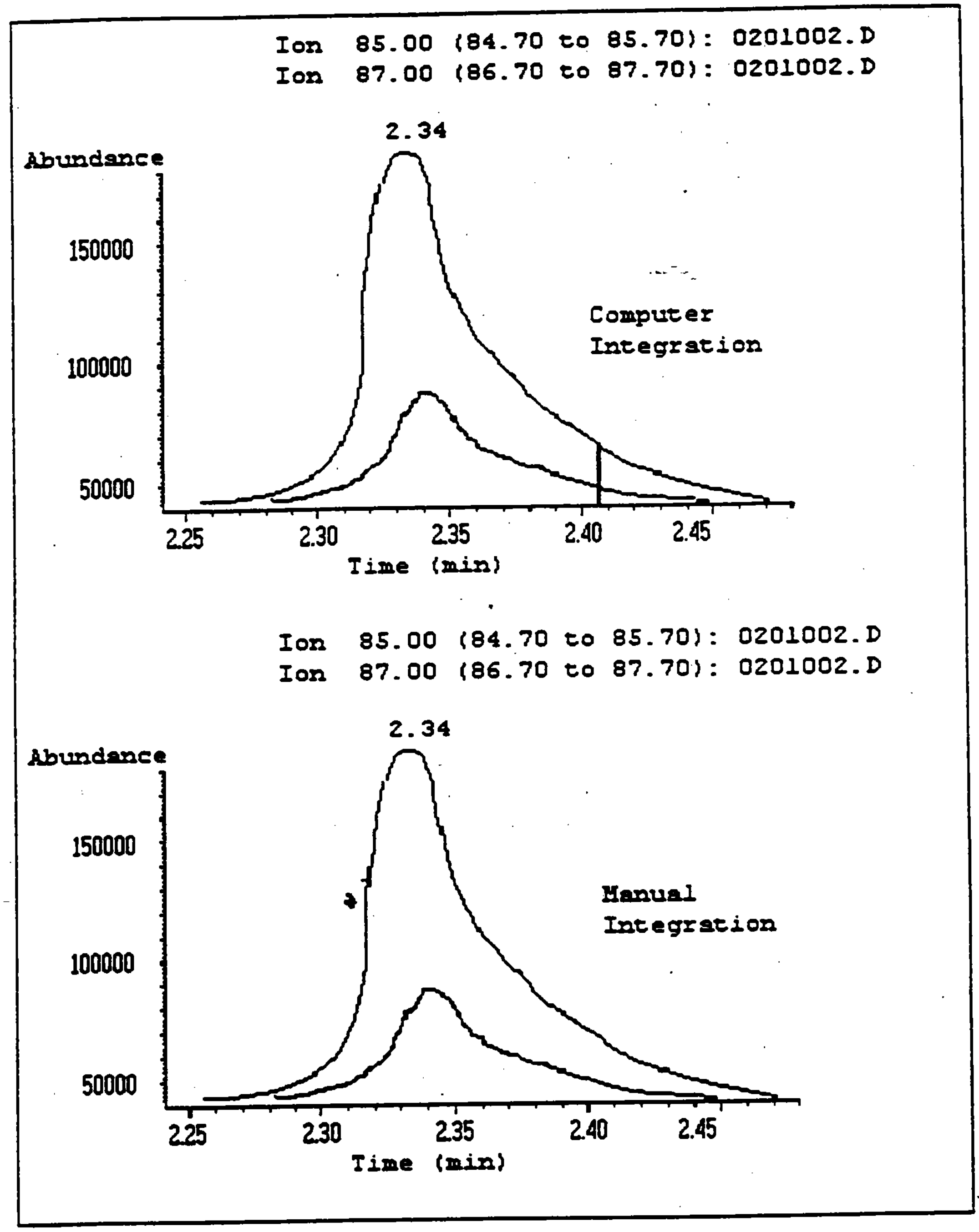
Attachment 1



Allowable Integration

Split Peaks

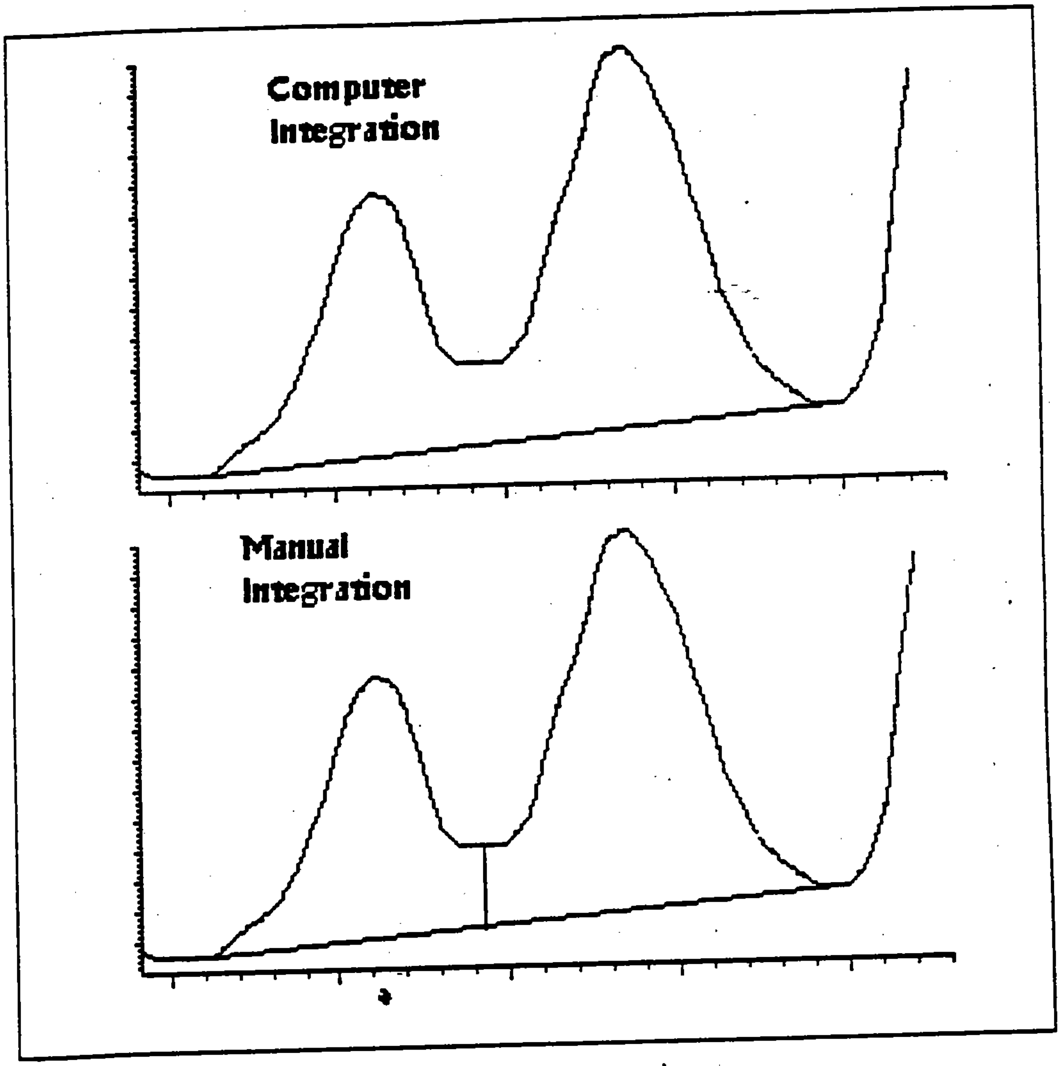
Attachment 2



Allowable Integration

Tailing

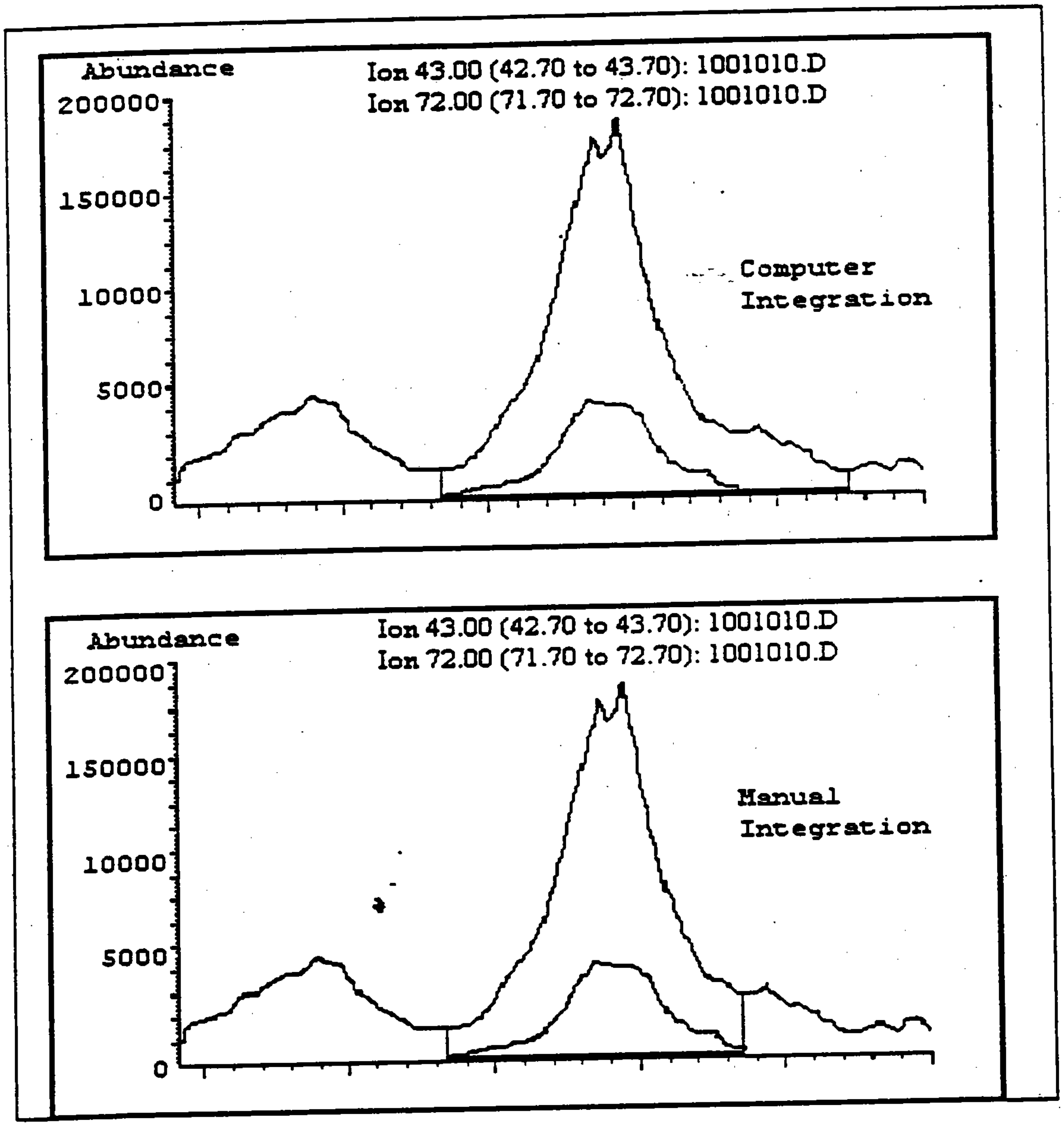
Attachment 3



Allowable Integration

Retention Time Shift - Mis-Identification - Merged

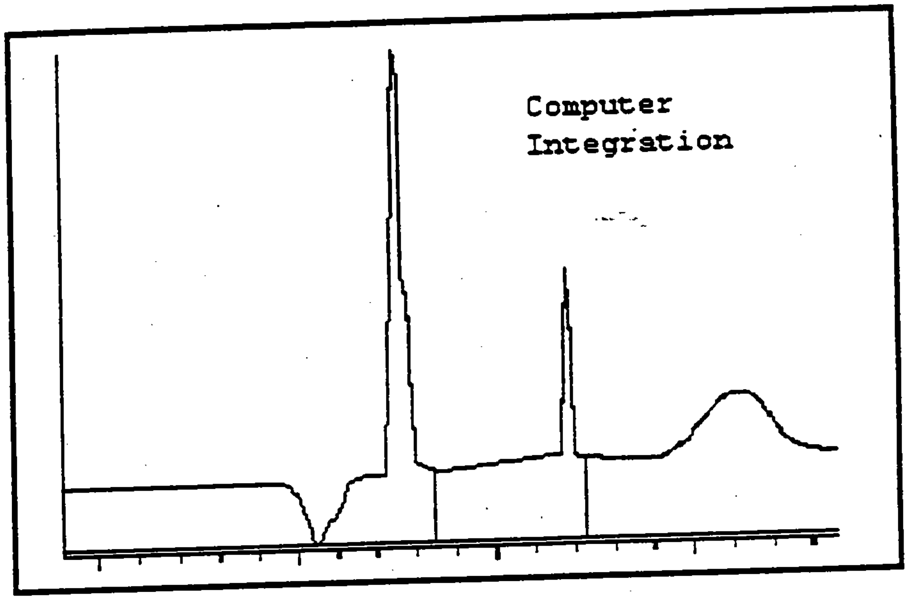
Attachment 4

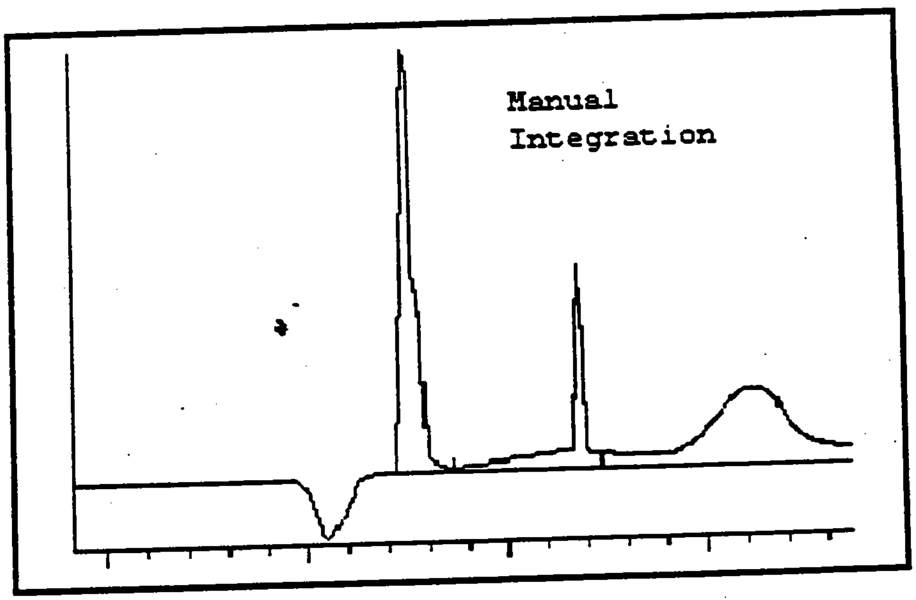


Allowable Integration

Secondary or Qualifier Ions

Attachment 5

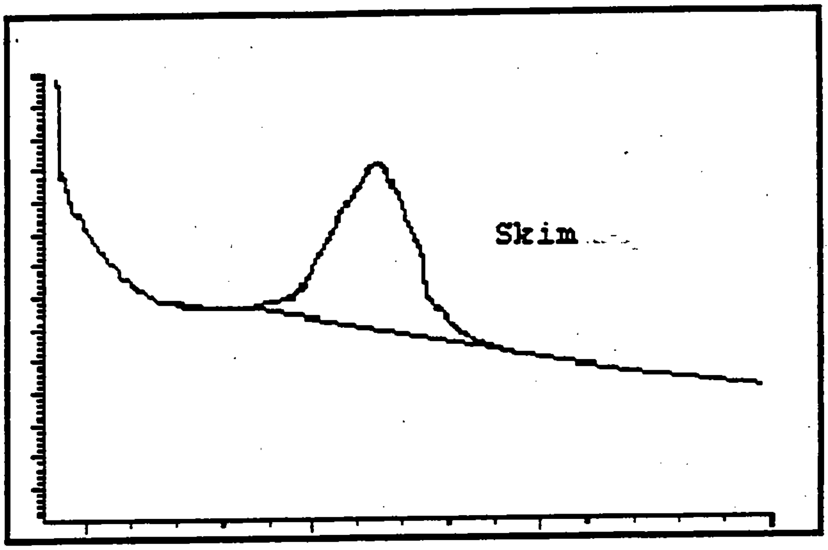


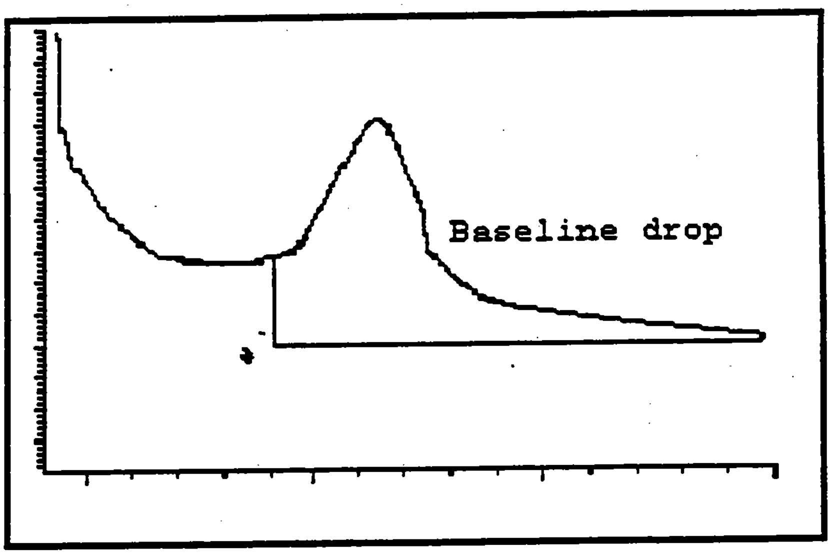


Allowable Integration

Baseline Shift

Attachment 6

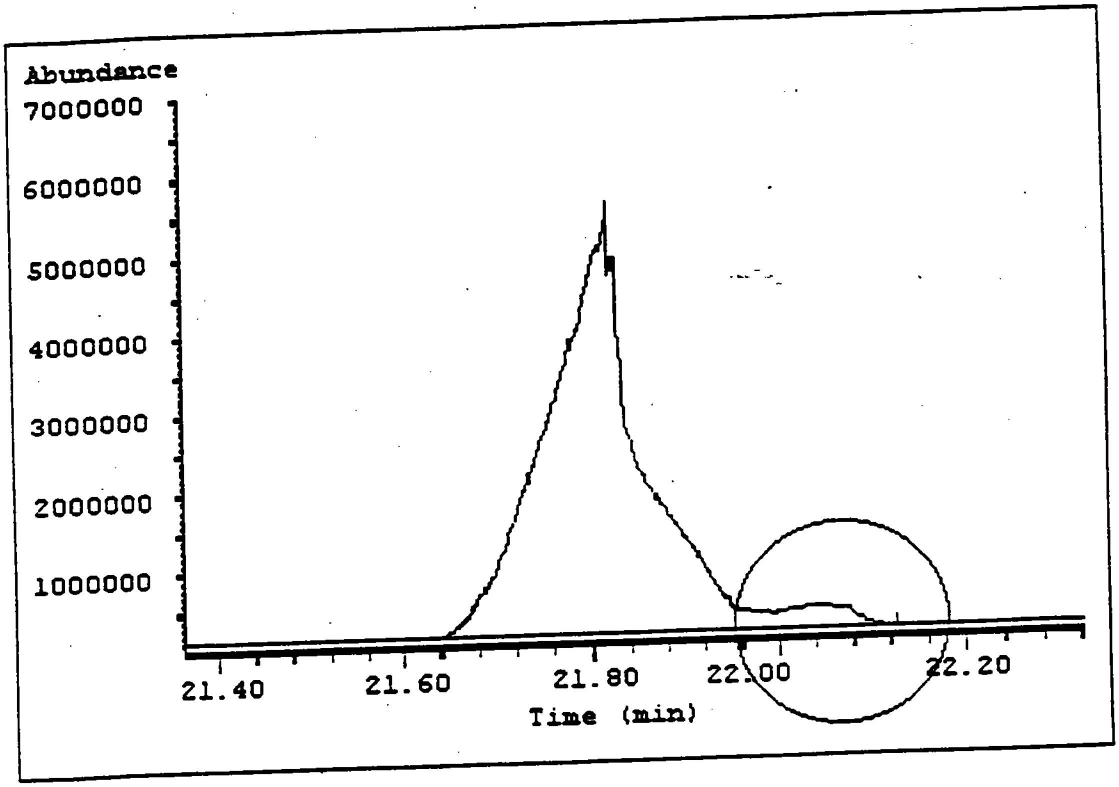




Allowable Integration

Skimming vs Baseline Drop

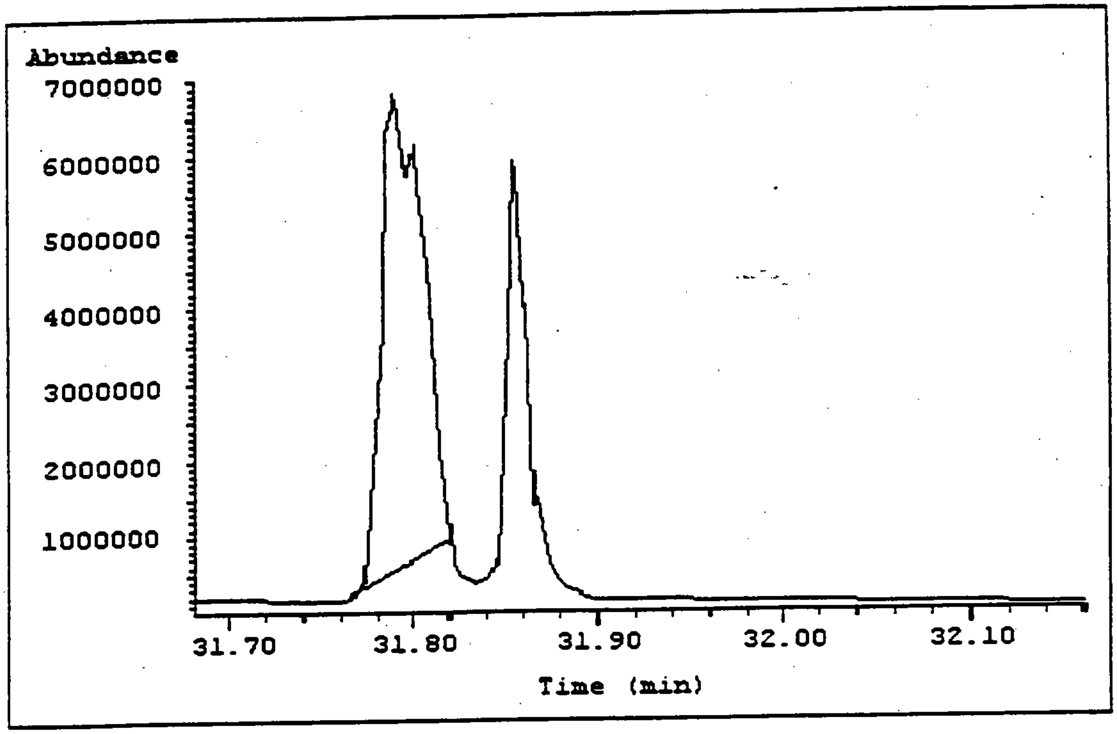
Attachment 7



__Improper__ Integration

Adding Area by Including Other Peaks

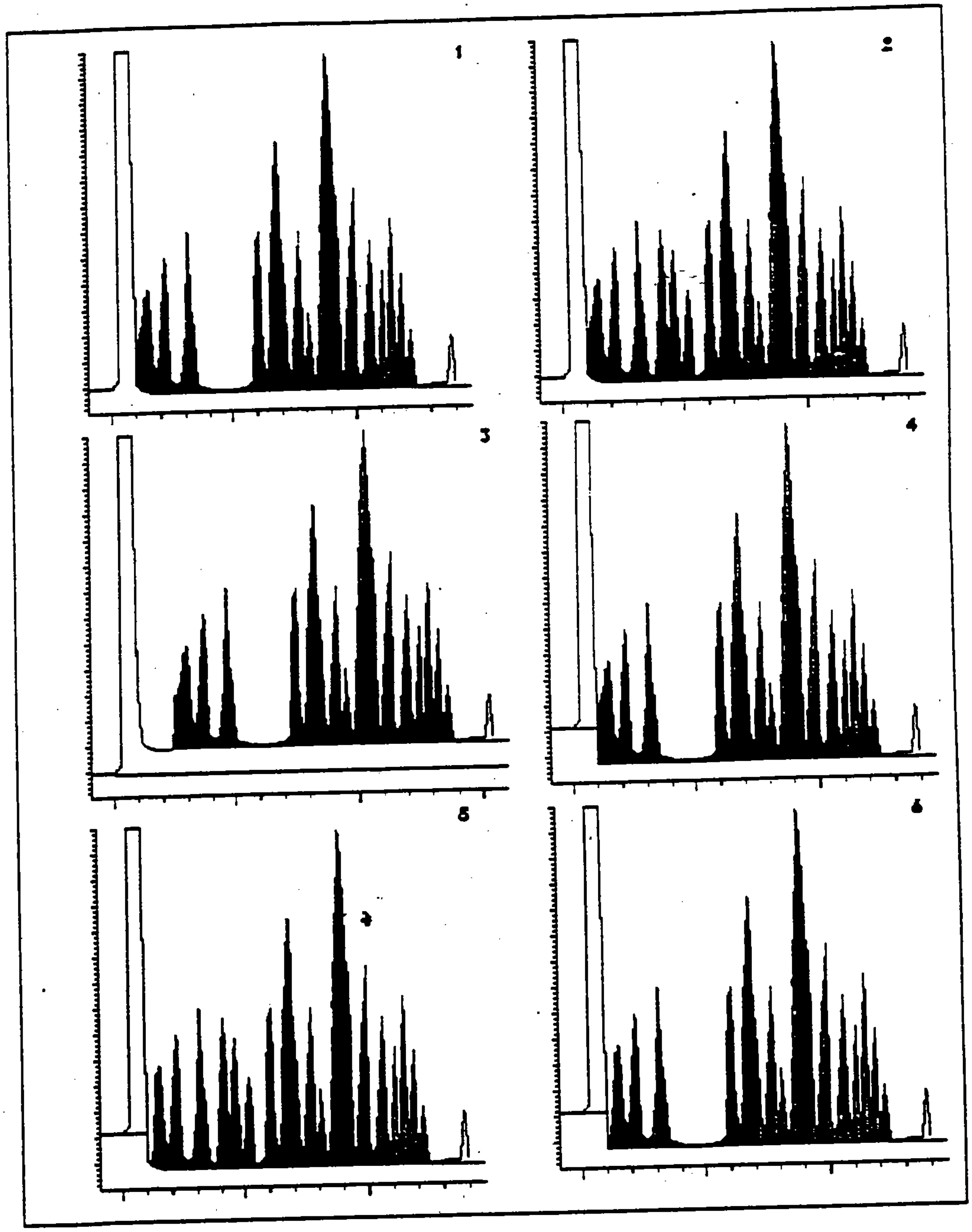
Attachment 8



Improper Integration

Baseline Moved To Decrease Peak Area

Attachment 9



Acceptable Methods of Integration
Grouped Peaks

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

Number	Date
1	4/10/91
2	9/11/91
3	4/26/94
4	4/09/97
5	5/12/98

Written by: Harry Romberg
Harry Romberg, Quality Assurance Officer
Date: 6-2-98

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 6-2-98

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD and TOC waste and soil samples. This SOP only covers handling of the waste from the point of collection.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the techniques described. As part of their training for analytical tasks which generate related wastes, each analyst must be trained to properly dispose of the waste or to the consolidate it at the appropriate collection point.

1.1.3 This SOP generally does not cover handling of the waste up to the point of disposal.

2. Equipment List

2.1 Equipment

2.1.1 The equipment necessary to properly dispose of laboratory wastes varies with the type of waste. In general, an appropriate container, packing material, and safety equipment (including clothing, eye wear, and respirators) is required.

3. Safety precautions

3.1 Safety Precautions

3.1.1 Solvent wastes may contain materials flammable at room temperature or lower. Caution should be taken to avoid flames and sparks when in the presence of or handling these wastes.

3.1.2 COD and TOC wastes may contain materials which will burn the skin, eyes, and/or mucous membranes if improperly handled. Precautions should be taken to avoid accidental contact.

3.1.3 All wastes may contain materials which can have both known and unknown long-term health effects. COD and TOC wastes, for instance, contain high levels of mercury salts. Direct contact should be avoided through the use of proper clothing and eye wear, even if no immediate danger is obvious. In the case of volatile solvents and other materials, handling should be done in a well-ventilated area and the exposure to vapors minimized. Where strong fumes are unavoidable, a carbon-filter or other respirator should be worn.

3.1.4 All people who handle waste products or the original reagents should be aware that the laboratory provides safety equipment and has a file containing Material Safety Data Sheets (MSDSs) on all laboratory chemicals in support of OSHA and other safety programs.

4. Operation procedures

4.1 Operations Appropriate to All Collection Areas

4.1.1 Land Disposal Restriction Forms (LDRs), manifests and other paperwork are not extensively discussed in this SOP because the disposal vendor deals with this aspect of the paperwork. It will only be necessary for the person who will be asked by the vendor to sign these forms (usually QA) to check that the information on the forms is accurate and to sign the form.

4.1.2 All waste requiring a Hazardous Waste disposal sticker and manifest will be marked with one of two EPA Hazardous Waste Site numbers. All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

4.1.3 The Hazardous Waste Sticker must be labeled with the proper DOT shipping name, even though the disposal company will usually replace the label before shipping. The proper shipping names are listed below in the applicable sections of this SOP.

4.1.4 All collection drums **must** be marked with an appropriately filled out Hazardous Waste sticker (see Appendix A). It is only necessary for Laucks staff to fill in the date that collection was **started** and the contents of the drum in the appropriate space. Hazardous Waste cannot be accumulated for longer than 90 days before it must be disposed. Therefore, do **not** mark the date on the drum until collection is started so as to maximize the allowable time until disposal. This sticker will be replaced by the transporter when they arrive to transport the waste to an approved disposal facility. The replacement sticker will contain all of the information required for transport and disposal.

4.1.5 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive or flammable nature (see Appendix B).

4.1.6 Once a material has been designated as waste and disposed into the designated drum, that drum must not be stored for longer than 90 days from the point that collection was **started**. This is rarely of concern at Laucks because transport is generally scheduled for most wastes within much less time than the required maximum storage time.

4.1.6.1 The one variation from the above rule is the TOC waste drum. This drum is not a satellite collection point but is actually the catch drum for the waste directly from the instrument. It will be disposed as soon as possible after it has reached capacity.

4.1.7 When collection drums are full or the 90-day limit is approaching, the Quality Assurance (QA) Department must be notified. The preferred lead-time for pickup is 10 working days so QA should actually be contacted 80 days after collection is initiated. This department, at the time of this writing, is responsible for contacting the appropriate approved transporter and insuring proper disposal takes place.

4.1.8 All questions or concerns regarding hazardous waste operations should first be directed to QA who will determine the appropriate course of action.

4.2 Mixed Solvent Waste

4.2.1 This waste stream is primarily composed of methylene chloride with some acetone and hexane and potentially small quantities of other solvents or dissolved products. The collection point for all of this waste is in the 921 facility (Extractions) solvent locker.

4.2.2 Small, 5 gallon or less containers of other mixed solvent waste may be collected as satellite accumulation units in the inorganics or organics instrument preparation areas but these **must** be transported to the primary drums in Extractions when full. Satellite accumulation containers must be kept closed when not in use and must be marked with the words "Hazardous Waste" or with other words that identify the contents of the container. This will most conveniently be done by using a blank Hazardous Waste Sticker.

4.2.3 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of "**Waste Flammable Liquids**".

4.2.4 Although methylene chloride is non-flammable, other components of these waste drums may be highly flammable. Thus, all of the waste solvent containers must be labeled as flammable.

4.2.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.6 When 3 or more full 55 gallon drums of this waste have been accumulated or 80 days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.7 At the time of this writing, Laucks uses Laidlaw Environmental as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis.

4.3 Chemical Oxygen Demand (COD) Waste

4.3.1 The primary constituents of this waste are sulfuric acid, water, mercury, silver, and chromium (both tri- and hexavalent). The collection point for this waste is in the inorganics area where CODs are analyzed. These analyses are conducted in small pre-packaged tubes. The reacted tubes are not considered to be waste until they are poured out of the tubes into a collection container.

4.3.2 Collection containers must be labeled with a Hazardous Waste sticker as previously noted. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Corrosive Liquids, Acidic, Inorganic**".

4.3.3 In addition to the hazardous waste sticker, these containers should be labeled as corrosive with the appropriate sticker as previously noted.

4.3.4 The waste not held for more than 90 days from initial collection (after pouring from the reaction tubes) until transportation for disposal. After 80 days have passed since the beginning of collection, QA must be contacted to arrange for transport and disposal within the allowable timeframe.

4.3.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.3.6 At the time of this writing, Laucks uses Laidlaw Environmental as the facility of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.4 Total Organic Carbon (TOC) Waste

4.4.1 The primary constituents of this waste are mercury, potassium persulfate, nitric acid, and water. This waste is collected directly from the instrument into a waste container beneath the instrument.

4.4.2 As this is a continuous process, Laucks does not begin the 90 day clock before disposal is required until this container is full. However, the container must be marked with a corrosive sticker. The Hazardous Waste sticker, in this case, must be dated as soon as the container is full and affixed at that time. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Corrosive Liquids, Acidic, Inorganic**".

4.4.3 At least 2 inches of headspace must be left between the top of the liquid and the top of the container to allow for expansion.

4.4.4 As soon as the container is full, the QA department must be notified to arrange for disposal. This waste stream will generally not be held in storage for very long after collection.

4.4.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.5 Soil Sample Disposal

4.5.1 State law allows a laboratory to store samples and other materials indefinitely, until they are considered waste and disposed. After that time, from the date of first accumulation, a 90 day timeframe is allowed before disposal must occur. Thus, soils should not be disposed of until enough have been accumulated to fill at least one 55 gal. drum.

4.5.2 Each drum used for soil waste disposal must be clearly marked with an identifying number which will be used to track which drum contained which samples. When samples are signed-out from their storage areas for disposal, the log sheet must be appropriate marked with the assigned drum number. This will enable the laboratory to track which samples were disposed in which drum.

4.5.2.1 The drums should be marked with a year, location from which they originate, and sequential number. Thus drums for which accumulation began in 1998 from the extractions laboratory would be marked 98-921-01. The -01 being a sequential number that would be incremented with each additional drum -02, -03, etc. throughout 1998. A drum from the main lab would be designated 98-940-01, etc.

4.5.2.2 When samples are transferred from the storage locations to the drums. the Secure Storage Custody Log must be marked to indicate into which drum they were disposed. This should include any bottle identifiers, if necessary to identify just what was disposed. Thus, it will be necessary for personnel disposing of samples to check the drums to make sure there is enough room for the designated samples. Soil samples will generally have their lids removed and disposed in the regular garbage. The jar and all will then be disposed in the waste drum. If the lids themselves contain client identifying marks or locations or have significant amounts of adhering material (oil, etc.) which cannot be readily dumped into the drum the lid will also be disposed into the waste drum.

4.5.2.3 When the drums are disposed, it will be necessary for the laboratory representative who signs the manifest to mark the drum identity on the manifest, although this only needs to be on the laboratory copy if the transporter does not want this information to appear on their copy of the record.

4.5.3 QA must be notified 80 days after accumulation has begun in order to arrange for disposal in a timely manner. If samples are not disposed until there is enough to fill a drum, this timeframe is not of major concern because there are always Hazardous Waste pickups scheduled within any 90 day time period.

4.5.4 The only stickers these drums must have is the Hazardous Waste sticker with the date accumulation was started clearly marked. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Environmentally Hazardous Substances**".

4.5.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. This vendor incinerates these soils prior to landfilling which should dispose of any organic materials, including labels, oily material and other hazardous organic substances.

4.6 PCB Oil Waste Disposal

4.6.1 Laucks no longer analyzes many oil samples for PCBs. Thus, this is a very small and infrequent waste stream. However, discussion is presented here in order that there be some documented course of action when it is necessary to dispose of these materials.

4.6.2 All oil samples which are analyzed for PCBs or otherwise known to contain PCBs are treated as PCB oils. No effort is made to distinguish those that actually do contain PCBs.

4.6.3 These oils are accumulated in a 5 gal. drum located in the Extractions laboratory warehouse. This metal drum is stored inside of the lower half of a cut-off plastic 55 gal. drum which fulfills the federal requirements for secondary containment during storage.

4.6.4 When a full drum has been accumulated, Eastern Electric is contacted for pickup and disposal. A signed receipt must be obtained as proof of disposal. Eastern Electric sends a manifest in subsequent mail within 35 days of waste pick-up and must also send a certificate of disposal within 30 days after the actual disposal date.

4.6.5 No annual report to the Department of Ecology is required because the level of PCBs is considered so high as to fall outside of the state's responsibility to monitor. At such levels the federal government regulates the disposal under TSCA. For this reason, it also does not fall within the federal requirement for RCRA governed waste disposal within 90 days. Eastern Electric is responsible for filing appropriate reports. TSCA regulations require that manifests and certificates of disposal be kept on file for a minimum of 3 years.

5. Reports

5.1 Disposal Paperwork

5.1.1 Our current vendor produce all of the required paperwork and insure all of the appropriate container markings (stickers, etc.) are in place prior to shipment. Since Laucks' waste streams are consistent from time to time, our vendors already have the information required to properly fill out the paperwork and Hazardous Waste stickers.

5.1.1.1 The paperwork includes the manifests, land disposal restriction forms and other shipping paperwork. Thus the only requirements of the laboratory are to insure the paperwork is accurate and to sign the appropriate forms.

5.1.2 After the waste has been transported to the disposal or accumulation facility, a signed manifest is returned to the laboratory. This is kept with the permanent record.

5.1.3 All certificates of disposal later provided by the disposal vendor are also associated with any waste shipment and kept with the permanent record.

5.1.4 All records are retained for at least 5 years from the date of shipment of the waste.

5.2 Annual Reporting Requirements

5.2.1 The laboratory must file an annual report with the Washington Department of Ecology (WDOE) for legal and tax purposes. This report is due on March 1 each year. Reports are filed for both the 940 and the 921 facilities (both EPA ID numbers). All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

5.2.1.1 The only exception to this reporting requirement is the reporting of the PCB waste oil which is a federally regulated waste and is thus not reported to the WDOE.

5.2.2 The format of this report is defined by WDOE in books provided to the laboratory several months in advance of the due date. Details of this report are not provided in this SOP.

5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update in September of each year. This report is more loosely defined and the only major requirement is that it be filed. Details of this report are not part of this SOP.

Appendix I

Hazardous Waste Sticker

See directions in this SOP for proper filling out of this sticker.

HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR
PUBLIC SAFETY AUTHORITY, OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

PROPER D.O.T.
SHIPPING NAME__________________________ UN OR NA#________

GENERATOR INFORMATION:

NAME______________________________________

ADDRESS___________________________________

CITY__________________________ STATE__________ ZIP________

EPA
ID NO.____________________ EPA
WASTE NO.____________________

ACCUMULATION
START DATE____________________ MANIFEST
DOCUMENT NO.____________________

HANDLE WITH CARE!

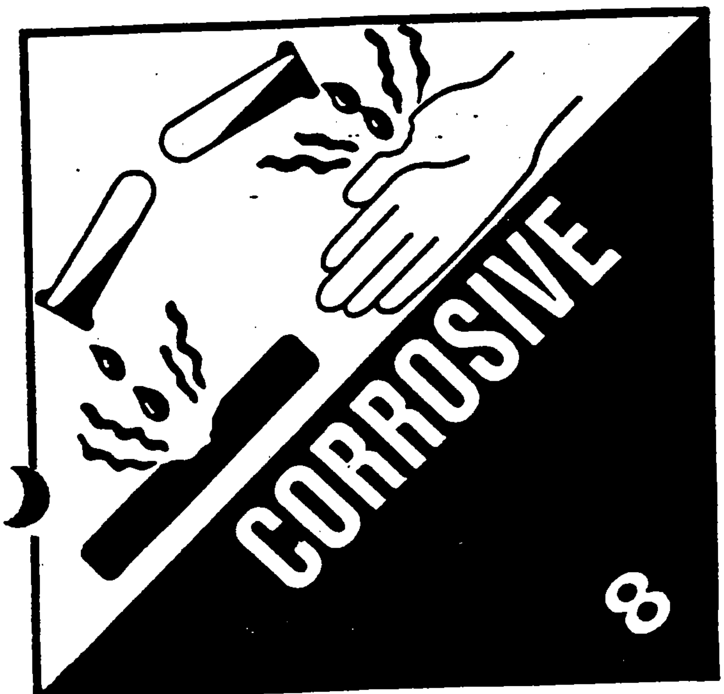
CONTAINS HAZARDOUS OR TOXIC WASTES

STYLE WM-6

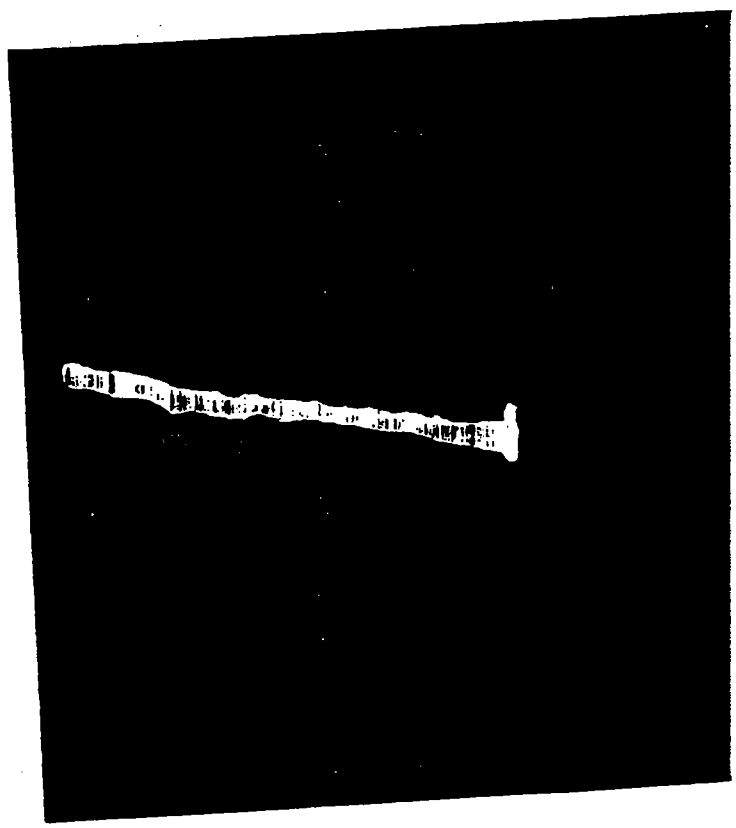
Printed by LABELMASTER, Div. of AMERICAN LABELMARK CO., INC., CHICAGO, IL 60646

Appendix II

Corrosive and Flammable Stickers



8-HML Published by J. J. KELLER & ASSOCIATES, INC. Neenah, WI 54956-0368 (414)722-2848



Extraction Method for Base, Acid and Neutral Compounds in Soil
(8270C by 3550B)

Method # LTL-3100

April 28, 1998

UNCONTROLLED

Revision Number: #11

Written by: B. O'Riell Date: 4-28-98

Reviewed by: Harry Romberg Date: 4-28-98

Approved by: Kathy E Kreps Date: 5/1/98

1. **PURPOSE** - In this method base, acid and neutral compounds are extracted from neutral soils with a mixture of methylene chloride and acetone. The extract is dehydrated, concentrated in a Kuderna-Danish (K-D) apparatus and GPC cleaned prior to analysis by GC Mass Spectrometer. This method provides for soils of low and medium levels of contamination.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a number of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples, by their very nature may contain significant levels of hazardous materials. It is incumbent on each analyst to exercise due care and caution executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents are to be AR grade or better.

3.2. All solvents are to be distilled in glass, unless otherwise noted.

3.3. The following special reagents should be prepared:

3.3.1. 1:1 methylene chloride/acetone (v/v)- prepared by mixing equal volumes of the solvents.

3.3.2. Anhydrous sodium sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.3. Surrogate solution prepared in methanol:

Base/Neutrals	Nitrobenzene-d5	200 ug/ml
	p-Terphenyl-d14	200 ug/ml
	2- Fluorobiphenyl	200 ug/ml
	1,2-Dichlorobenzene-d4	200 ug/ml
Acids	Phenol-d5	300 ug/ml
	2,4,6-Tribromophenol	300 ug/ml
	2-Fluorophenol	300 ug/ml
	2-Chlorophenol-d4	300 ug/ml

3.3.4. Matrix spiking solution prepared in methanol.

Base/Neutrals	1,2,4-Trichlorobenzene	100 ug/ml
	Acenaphthene	100 ug/ml
	2,4-Dinitrotoluene	100 ug/ml
	Pyrene	100 ug/ml
	n-Nitroso-di-n-propylamine	100 ug/ml
	1,4-Dichlorobenzene	100 ug/ml
Acids	Pentachlorophenol	150 ug/ml
	Phenol	150 ug/ml
	2-Chlorophenol	150 ug/ml
	4-Chloro-3-methyl phenol	150 ug/ml
	4-Nitrophenol	150 ug/ml

4. EQUIPMENT

4.1. Heat Systems Ultrasonic Processer - Model XL2020, 550 watts - Maintain per manufacturer's instructions.

4.1.1. 3/4 inch titanium horn (#208) for low concentrations

4.1.2. 1/8 inch tapered titanium microtip (#419) for medium concentrations

4.2. Analytical Biochemical Laboratories, Inc. (ABC) Model 1002B or Model 1000 Gel-Permeation Chromatography (GPC).

4.3. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.4. Standard laboratory glassware to include:

4.4.1. 8 ounce extractions jars

4.4.2. 500 ml Fleakers

4.4.3. K-D apparatus: 500 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.5. All glassware to be rinsed as follows, prior to use:

4.5.1. Technical grade acetone (if the glassware is wet).

4.5.2. Triple rinsed with methylene chloride.

4.6. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

NOTE: It is necessary to make an intuitive decision as to which level extraction procedure to follow. If the soil appears clean with little odor, do a low level extraction. If the soil has appreciable odor, is tar-like, partially miscible, or has an oily appearance, prep both a low and a medium level extraction. Totally miscible samples may only have a medium level extraction. Remember it is important that holding times be met. If you are in doubt, prep and store a medium level sample simultaneously.

6.1. Sample Extraction - Low Level Soils

6.1.1. Mix sample thoroughly in its original container if there is space available (otherwise mix in solvent rinsed aluminum tray).

6.1.2. Weigh 30.0 grams of soil (wet weight) into an extraction bottle.

6.1.3. Prepare two additional aliquots of a sample if Quality Control is required.

6.1.4. Add 30 to 60 grams sodium sulfate and mix well to give the soil a sandy texture.

6.1.5. Prepare a blank and a blank spike with 60 grams sodium sulfate.

6.1.6. Pipet 500 ul surrogate solution to each bottle.

6.1.7. Pipet 500 ul matrix spiking solution to each of the QC bottles.

6.1.8. Add 100 ml 1:1 methylene chloride/acetone to each bottle.

6.1.9. Sonicate the bottles for 3 minutes using the sonic horn, set at 50% duty cycle and full output (10).

6.1.10. Centrifuge the samples for 20 minutes at 2000 rpm if necessary to achieve a partition.

6.1.11. Decant off and collect the supernatant.

6.1.12. Repeat from step 6.1.8. two additional times, combining all of the extracts.

6.2. Solvent dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from step 6.1.12. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent Evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 12 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml and remove the apparatus from the water bath. Cool to room temperature.

6.3.4. Rinse joint and remove snyder column. Allow rinse solvent to drain into ampule.

6.3.5. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of solvent.

6.3.6. Reduce the extract volume to less than 8 ml on a nitrogen blowdown. Transfer extract to a 16 x 100 mm culture tube, and adjust to 10.0 ml intermediate volume (as compared to a measured volume) with methylene chloride.

6.3.7. At this point the extract will be GPC cleaned -- see Method # LTL-3692.

6.3.8. K-D the GPC cleaned extract. Reduce the extract to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of ampule several times during blowdown.

6.3.9. Transfer the extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methylene chloride.

6.3.10. Label the extract and deliver to 940.

6.3.11. Complete all paperwork and bench sheet. Bench sheet to include the date of GPC, date and time of transfer to 940 and extract location. Clip the T-Card on the folder and place in GC/MS room extraction folder box. The file folder color will be blue and the blank name will be ____MSV.SL_.

6.4. Sample Extraction - Medium Level Soils

6.4.1. Weigh 2.00 gram soil into a silanized scintillation vial.

6.4.2. Prepare two additional aliquots of a sample if Quality Control is required.

6.4.3. Add two grams sodium sulfate to all sample vials.

6.4.4. Prepare a blank and blank spike consisting of two grams of sodium sulfate.

6.4.5. Pipet 500 ul of the surrogate into each vial.

6.4.6. Pipet 500 ul matrix spiking solution into each of the QC vials.

6.4.7. Add 9.5 ml methylene chloride to each tube and 9.0 ml to each QC vial.

6.4.8. Sonicate the vials for 2 minutes using the sonic horn (microtip), set at 50% duty cycle and half output (5).

6.4.9. Loosely pack a Monstr-pipet column 1/3 full (approx. 3 cm) with solvent cleaned glass wool. Add approx. 1 cm sodium sulfate.

6.4.10. Place a 16 x 100 mm culture tube under the column.

6.4.11. Pass the extract through the column. Collect a minimum of 8.0 mls.

6.4.12. At this point the extract will be GPC cleaned -- see Method # LTL-3692.

6.4.13. K-D the GPC cleaned extract. Reduce the extract to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of ampule several times during blowdown.

6.4.14. Transfer the extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methylene chloride.

6.4.15. Label the extract and deliver to 940.

6.4.16. Complete all paperwork and bench sheet. Bench sheet to include the date of GPC, date and time of transfer to 940 and extract location. Clip the T-Card on the folder and place in GC/MS room extraction folder box. The file folder color blue and the blank name will be ____MSV.SM_.

7. **ANALYSIS TIME** - Based on extensive experience in the laboratory, it is anticipated that a single sample may be completed in about ten hours. If it is possible to batch similar samples, it is expected that about six samples could be completed in approximately 16 hours. About one and one half hours of hands on extractionist time will be required per sample. These approximations are based on the assumption that the samples are "average", and will not require additional time beyond normal operations.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846 (1996)

8270C, 3550B

Extraction Method for Ordnance Compounds in Soils (8330)

Method # LTL-3161

June 1, 1998

UNCONTROLLED

Revision Number: #3

Written by: ______ Date: 6-1-98

Reviewed by: Harry Romberg Date: 6-1-98

Approved by: Kathy E Kreps Date: 6-1-98

1. **PURPOSE** - In this method the ordnance compounds are extracted from neutral soil with acetonitrile. The extract is salted-out, filtered and analyzed by HPLC.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2. All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. Ottawa Sand - prepared by soxhleting for 8 hours with methylene chloride, air dry for two hours in a hood, followed by two hours in a 100°C oven.

3.3.2. 0.045 M Calcium Chloride - prepared by dissolving 5.0 gm in 1000 ml of DIW.

3.3.3. Surrogate solution prepared in methanol:

1,2-Dinitrobenzene	400 ug/ml

3.3.4. Matrix spiking (MS) solution prepared in methanol:

1,3-Dinitrobenzene	40 ug/ml
2,4-Dinitrotoluene	40 ug/ml
2,6-Dinitrotoluene	40 ug/ml
HMX	40 ug/ml
RDX	40 ug/ml
Nitrobenzene	40 ug/ml
2-Nitrotoluene	40 ug/ml
3-Nitrotoluene	40 ug/ml
4-Nitrotoluene	40 ug/ml
Tetryl	40 ug/ml
TNT	40 ug/ml
1,3,5-Trinitrobenzene	40 ug/ml
2-amino-4,6-dinitrotoluene	40 ug/ml
4-amino-2,6-dinitrotoluene	40 ug/ml

4. EQUIPMENT

4.1. Ultrasonic bath

4.2. Disposable cartridge filters - 0.45 µm Teflon filter

4.3. Standard laboratory glassware to include:

4.3.1. 20 x 150 mm culture tubes

4.4. Volumetric measurements are to be made with graduated serological pipets or a calibrated fixed volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container if there is space available (otherwise mix in solvent rinsed aluminum tray).

6.1.2. Weigh approximately 20 grams into a labeled, large aluminium weighing tin. Place in hood and air dry for 48 hours or until a constant weight is attained.

6.1.3. Weigh 2.0 grams of air dried soil into a 20 x 150 mm culture tube.

6.1.4. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.5. Prepare a blank and blank spike with 2.0 grams Ottawa sand.

6.1.6. Pipet 100 ul surrogate solution to each culture tube.

6.1.7. Pipet 1000 ul matrix spiking solution to each of the QC culture tubes.

6.1.8. Add 9.9 ml acetonitrile to each sample culture tube and blank and 8.9 mls to QC and blank spike.

6.1.9. Sonicate the samples for 18 hours in a sonic bath packed with ice in the cold room.

6.2. Salting Out

6.2.1. Remove samples from ice bath and allow to come to room temperature.

6.2.2. Transfer 5.0 mls of supernatant to a 16 x 100 mm culture tube.

6.2.3. Add 5.0 ml calcium chloride solution to each sample.

6.2.4. Shake and let stand for 15 minutes.

6.2.5. Centrifuge each sample.

6.2.6. Filter through a 0.45μm PTFE syringe filter into 4 ml **amber** vial, discarding first 3 mls.

6.2.7. Label the extract and deliver to 940.

6.3.8. Complete all paperwork and bench sheet. Bench sheet to include PTFE filter lot #, date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be red and the blank name will be ____HOR.SM_.

7. **ANALYSIS TIME** - Based on experience in the laboratory, it is anticipated that a single sample may be completed in about 20 hours. If it is possible to batch similar samples, it is expected that about ten samples could be completed in approximately 24 hours. About one hour of actual hands on time will be expended by the extractionist. These approximate times are based on the assumption that the samples are "average", and will not require additional time beyond normal operations. Additional time must be allocated for samples which are very dirty or are extraordinary.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

8330 (1994)

Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Soil (8081A/8082 by 3550B)

Method # LTL-3302

November 29, 1999

Revision Number: #2

Written by: B. O [signature] Date: 11.29.99

Reviewed by: Harry Romberg Date: 12-13-99

Approved by: Kathy Orreps Date: 12-13-99

Controlled Document

No. 20 Assigned to: Tetra

1. **PURPOSE** - In this method organochlorine pesticides and polychlorinated biphenyls are extracted from neutral soil with a mixture of methylene chloride and acetone. The extract is dehydrated and concentrated in a Kuderna-Danish (K-D) apparatus. The extract is split prior to any cleanup step. The extracts are GPC and SPF florisil cleaned prior to analysis by GC. The PCBs receive an additional sulfuric acid cleanup.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples, by their very nature, may contain significant levels of hazardous materials. It is incumbent on each analyst to exercise due care and caution executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents are to be AR grade or better.

3.2. All solvents are to be distilled in glass, unless otherwise noted.

3.3. The following special reagents should be prepared:

3.3.1. 1:1 methylene chloride/acetone (v/v)- prepared by mixing equal volumes of the solvents.

3.3.2. 9:1 Hexane/acetone - prepared by adding 10.0 ml acetone to 90.0 ml hexane.

3.3.3. Anhydrous sodium sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.4. Florisil SPE column check solution prepared in acetone:

2,4,5-Trichlorophenol	0.100 ug/ml
Gamma BHC	0.020 ug/ml
Heptachlor	0.020 ug/ml
Endrin	0.040 ug/ml
4,4-DDD	0.040 ug/ml
4,4-DDT	0.040 ug/ml
Methoxychlor	0.200 ug/ml
Alpha BHC	0.020 ug/ml
Dieldrin	0.040 ug/ml
Alpha Endosulfan	0.020 ug/ml
Tetrachloro-m-xylene	0.020 ug/ml
Decachlorobiphenyl	0.040 ug/ml

3.3.5. Surrogate solution prepared in acetone:

2,4,5,6-Tetrachloro-m-xylene	1.0 ug/ml
Decachlorobiphenyl	1.0 ug/ml

3.3.6. Pesticide/PCB matrix spiking solution prepared in acetone:

Gamma-BHC	2.5 ug/ml
Heptachlor	2.5 ug/ml
Aldrin	2.5 ug/ml
Arochlor 1260	25.0 ug/ml

4. EQUIPMENT

4.1. Heat Systems Ultrasonic Processer - Model XL2020, 550 watts - Maintain per manufacturer's instructions.

4.1.1. 3/4 inch titanium horn (#208)

4.2. Analytical Biochemical Laboratories, Inc. (ABC) Model 1002B or Model 1000 Gel-Permeation Chromatography (GPC)

4.3. Organomations Assoc., Inc. - N-EVAP (nitrogen evaporator), Model 112

4.4. Analytechem International - Vac Elaut SPS24 (for SPE florisil columns)

4.5. Florisil SPE cartridges with Teflon frits (1000 mg)

4.6. Standard laboratory glassware to include:

4.6.1. 8 ounce extraction bottle

4.6.2. 500 ml Fleakers

4.6.3. K-D apparatus: 500 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.7. All glassware to be rinsed as follows, prior to use:

4.7.1. Technical grade acetone (if the glassware is wet).

4.7.2. Triple rinsed with methylene chloride.

4.8. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container if there is space available (otherwise mix in solvent rinsed aluminum tray).

6.1.2. Weigh 30.0 grams of soil (wet weight) into a extraction bottle.

6.1.3. Prepare two additional aliquots of a sample if Quality Control is required.

6.1.4. Add 60 grams sodium sulfate and mix well to give the soil a sandy texture.

6.1.5. Prepare a blank and a blank spike with 60 grams sodium sulfate.

6.1.6. Pipet 200 ul surrogate solution to each bottle.

6.1.7. Pipet 200 ul matrix spiking solution to each of the QC bottles.

6.1.8. Add 100 ml 1:1 methylene chloride/acetone to each bottle.

6.1.9. Sonicate the bottles for 3 minutes using the sonic horn, set at 50% duty cycle and full output (10).

6.1.10. Centrifuge the samples for 20 minutes at 2000 rpm if necessary to achieve a partition.

6.1.11. Decant off and collect the supernatant.

6.1.12. Repeat from steps 6.1.8. two additional times, combining all of the extracts.

6.2. Solvent dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from step 6.1.12. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 12 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml, remove the apparatus from the water bath and cool to room temperature.

6.3.4. Rinse joint and remove snyder column. Allow rinse solvent to drain into ampule.

6.3.5. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of solvent.

6.3.6. Reduce the extract volume to less than 8 ml on a nitrogen blowdown. Transfer extract to a 16 x 100 mm culture tube, and adjust to 10.0 ml intermediate volume (as compared to a measured volume) with methylene chloride.

NOTE: In instances where sample extracts are being prepared for PCB only (8082), and the GPC step is being omitted prior to acid cleanup, the solvent exchange step (see 6.3.8.1.) must be tested. Take 1 ml extract, 1 ml sulfuric acid and vortex. Check for volume changes between layers. If exchange is complete, proceed to 6.4.

6.3.7. At this point the extract will be GPC cleaned -- see Method # LTL-3692.

6.3.8. K-D the GPC cleaned extract to 4-5 ml and exchange into hexane as follows:

6.3.8.1. Add 2-3 ml hexane through the top of the snyder column while the ampule is still immersed in the water bath. Reduce to 4-5 ml.

6.3.8.2. Repeat 6.3.8.1. two additional times.

6.3.9. Remove the apparatus from the water bath and cool to room temperature.

6.3.10. Rinse joint with hexane and remove snyder column. Allow rinse solvent to drain into ampule.

6.3.11. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of hexane.

6.3.12. Reduce the extract to less than four ml in a nitrogen blowdown. Transfer extract to a 16 x 100 mm culture tube and adjust to a final volume of 5.0 ml (as compared to a measured volume) in hexane.

6.4. Sulfuric Acid Cleanup (PCBs only)

6.4.1. Transfer a two ml aliquot from the 5.0 ml final volume (step 6.3.12.) to a 16 x 100 mm culture tube.

6.4.2. Add 2.0 mls concentrated sulfuric acid and vortex for 30 - 60 seconds.

6.4.3. Let stand for a few minutes to allow layers to separate. May be centrifuged.

6.5. Florisil Cleanup

6.5.1. Attach the vacuum manifold to the vacuum pump with a trap in between. Place a labeled 16 x 100 mm culture tube in the proper collection slot. Secure the manifold with the straps and move the manifold to the waste position. Adjust the vacuum pressure in the manifold to between five and ten pounds of pressure.

6.5.2. Place one florisil cartridge into the vacuum manifold.

6.5.3. Start the vacuum and prewet the cartridge with hexane/acetone (9:1), by passing at least 5 ml through the cartridge. Do not allow the cartridge to go dry after wetting.

6.5.4. Release the vacuum, and move the manifold to the collect position.

6.5.5. Add 1.0 ml extract from step 6.3.12. (Pesticides) or from step 6.4.3. (PCBs). to the top frit of the florisil cartridge. (Store remaining 4.0/3.0 ml of extract.)

6.5.6. Restore the vacuum, and elute the column with 8-9 ml of hexane/acetone (9:1). Allow the cartridge to go dry.

6.5.7. Release the vacuum, and move the manifold to the waste position. Remove and discard the used florisil cartridge.

6.5.8. Repeat from step 6.5.2. for additional extracts.

6.5.9. Reduce the extract volume to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of culture tube several times during blowdown.

6.5.10. Transfer extract to a 1.8 ml vial. The volume is then adjusted to 1.0 ml (as compared to a measured volume) in hexane.

6.5.11. Label the extract and deliver to 940.

6.5.12. Complete all necessary paperwork and bench sheet. Bench sheet to include the date of GPC, florisil lot number, date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in the GC room extraction folder box. The file folder color will be purple and the blank name will be ____GPX.SL_.

7. **ANALYSIS TIME** - Based on experience in the laboratory, it is anticipated that a single sample may be completed in about 12 hours. If it is possible to batch similar samples it is expected that about seven samples could be completed in approximately 20 working hours. About four and a half hours of hands on time will be required of the extractionist. These approximations are based on the assumption that the samples are "average", and will not require additional time beyond normal operations. Additional time must be allocated for samples which are very dirty or are extraordinary, such as tissues or vegetable matter.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846, (1996).

8081A, 8082, 3550B.

Extraction Method for Polynuclear Aromatics in Soil
(8270C by 3550B)

Method # LTL-3450

November 29, 1999

Revision Number: #1

Written by: B. O Quiòe Date: 11-29-99

Reviewed by: Harry Romberg Date: 12-13-99

Approved by: Kathy Krebs Date: 12-13-99

Controlled Document
No. 20 Assigned to: Tetra

1. **PURPOSE** - In this method, polynuclear aromatics are extracted from neutral soils with a mixture of methylene chloride and acetone. The extract is dehydrated, concentrated in a Kuderna-Danish (K-D) apparatus and alumina cleaned prior to analysis by GC Mass Spectrometer. This method also includes low concentration (SIM).

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a number of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples, by their very nature may contain significant levels of hazardous materials. It is incumbent on each analyst to exercise due care and caution executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents are to be AR grade or better.

3.2. All solvents are to be distilled in glass, unless otherwise noted.

3.3. The following special reagents should be prepared:

3.3.1. 1:1 methylene chloride/acetone (v/v)- prepared by mixing equal volumes of the solvents.

3.3.2. Anhydrous sodium sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.3. Alumina, Neutral, Brockman Activity I - activate 60-325 mesh alumina by heating for 16 hours at 130° C.

3.3.4. Surrogate solution prepared in methanol:

1-Fluoronaphthalene	250 ug/ml
Fluorene-d10	250 ug/ml
Pyrene-d10	250 ug/ml

Low Concentration (SIM) surrogate solution prepared in methanol:

1-Fluoronaphthalene	5.0 ug/ml
Fluorene-d10	5.0 ug/ml
Pyrene-d10	5.0 ug/ml

3.3.5. Matrix spiking solution and Low Concentration (SIM) matrix spiking solution prepared in methanol:

Acenaphthene	50 ug/ml
Acenaphthylene	50 ug/ml
Anthracene	50 ug/ml

Benzo(a)anthracene	50 ug/ml
Benzo(a)pyrene	50 ug/ml
Benzo(b)fluoranthene	50 ug/ml
Benzo(ghi)perylene	50 ug/ml
Benzo(k)fluoranthene	50 ug/ml
Chrysene	50 ug/ml
Dibenzo(a,h)anthracene	50 ug/ml
Fluoranthene	50 ug/ml
Fluorene	50 ug/ml
Indeno(1,2,3-cd)pyrene	50 ug/ml
Naphthalene	50 ug/ml
Phenanthrene	50 ug/ml
Pyrene	50 ug/ml
2-Methylnaphthalene	50 ug/ml

4. EQUIPMENT

4.1. Heat Systems Ultrasonic Processor - Model XL2020, 550 watts - Maintain per manufacturer's instructions.

4.1.1. 3/4 inch titanium horn (#208).

4.2. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.3. Standard laboratory glassware to include:

4.3.1. 8 ounce extraction bottles

4.3.2. 500 ml Fleakers

4.3.3. K-D apparatus: 500 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.4. All glassware to be rinsed as follows, prior to use:

4.4.1. Technical grade acetone (if the glassware is wet).

4.4.2. Triple rinsed with methylene chloride.

4.5. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

4.6. Teflon thistle tubes

4.7. Disposable Monstr-pette (large pasteur pipets)

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

6.1. Sample Extraction:

6.1.1. Mix sample thoroughly in its original container if there is space available (otherwise mix in solvent rinsed aluminum tray).

6.1.2. Weigh 30.0 grams of soil (wet weight) into an extraction bottle.

6.1.3. Prepare two additional aliquots of a sample if Quality Control is required.

6.1.4. Add 30 to 60 grams sodium sulfate and mix well to give the soil a sandy texture.

6.1.5. Prepare a blank and a blank spike with 60 grams sodium sulfate.

6.1.6. Pipet 200 ul surrogate solution to each bottle.
(Low Conc.: 500 ul of low conc. surrogate solution.)

6.1.7. Pipet 500 ul matrix spiking solution to each of the QC bottles.
(Low Conc.: 50 ul of matrix spiking solution.)

6.1.8. Add 100 ml 1:1 methylene chloride/acetone to each bottle.

6.1.9. Sonicate the bottles for 3 minutes using the sonic horn, set at 50% duty cycle and full output (10).

6.1.10. Centrifuge the samples for 20 minutes at 2000 rpm if necessary to achieve a partition.

6.1.11. Decant off and collect the supernatant.

6.1.12. Repeat from step 6.1.8. two additional times, combining all of the extracts.

6.2. Solvent dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from step 6.1.12. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent Evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° ~~C,~~ with a long ampule immersed to a depth of 12 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml and remove the apparatus from the water bath. Cool to room temperature.

6.3.4. Rinse joint and remove snyder column. Allow rinse solvent to drain into ampule.

6.3.5. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of solvent.

6.3.6. Reduce the extract to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of ampule several times during blowdown.

6.4. Alumina Cleanup

6.4.1. Prepare a Monstr-pette column by plugging with a small amount of glass wool.

6.4.2. Pack column with a minimum of 3 gm. of activated alumina. Top with 1/2 cm of sodium sulfate.

6.4.3. Attach Teflon thistle tube and pre-elute column with 10 ml methylene chloride.

6.4.4. Add extract directly to top of the sodium sulfate and allow to elute onto the column.

6.4.5. Rinse the ampule twice with a small amount of methylene chloride and add to top of column.

6.4.6. Re-attach the Teflon thistle tube to the column and add a minimum of 10 ml methylene chloride

6.4.7. Elute into a 16 x 125 mm culture tube.

6.4.8. Reduce the extract volume to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of the culture tube several times during blowdown.

6.4.9. Transfer extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methylene chloride. (Low Conc.: Final volume adjusted to 1.0 ml. in methylene chloride.)

6.4.10. Label the extract and deliver to 940.

6.4.11. Complete all paperwork and bench sheet. Bench sheet to include alumina lot #, the date of transfer to 940 and extract location. Clip the T-Card on the ~~folder~~ and place in GC/MS room extraction folder box. The file folder color will be blue and the blank name will be ____MPN.SL_.

7. **ANALYSIS TIME** - Based on extensive experience in the laboratory, it is anticipated that a single sample may be completed in about ten hours. If it is possible to batch similar samples, it is expected that about six samples could be completed in approximately 16 hours. About one and one half hours of hands on extractionist time will be required per sample. These approximations are based on the assumption that the samples are "average", and will not require additional time beyond normal operations.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846.

8270C (1996), 3550B (1996), 3610B (1996)

6.4.6. Re-attach the Teflon thistle tube to the column and add a minimum of 10 ml methylene chloride

6.4.7. Elute into a 16 x 125 mm culture tube.

6.4.8. Reduce the extract volume to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of the culture tube several times during blowdown.

6.4.9. Transfer extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methylene chloride. (Low Conc.: Final volume adjusted to 1.0 ml. in methylene chloride.)

6.4.10. Label the extract and deliver to 940.

6.4.11. Complete all paperwork and bench sheet. Bench sheet to include alumina lot #, the date of transfer to 940 and extract location. Clip the T-Card on the folder and place in GC/MS room extraction folder box. The file folder color will be blue and the blank name will be ____MPN.SL_.

7. **ANALYSIS TIME** - Based on extensive experience in the laboratory, it is anticipated that a single sample may be completed in about ten hours. If it is possible to batch similar samples, it is expected that about six samples could be completed in approximately 16 hours. About one and one half hours of hands on extractionist time will be required per sample. These approximations are based on the assumption that the samples are "average", and will not require additional time beyond normal operations.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846.

8270C (1996), 3550B (1996), 3610B (1996)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

Revision history:

Number	**Date**
6	3/18/99
5	6/19/96
4	12/28/95
3	4/05/95
2	6/05/90
1	4/24/90

Revised by: Pam Johnson
Pam Johnson, Sample Control
Date: 3/25/99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 3-25-99

Approved by: Harry Romberg
Harry Romberg, QA Officer
Date: 3-25-99

Controlled Document

No. 20 **Assigned to:** TETRA

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1. INTRODUCTION AND SCOPE

1.1 Introduction

1.1.1 This procedure is a description of sample receipt, sample log-in, and sample tracking when samples are logged into the laboratory's Laboratory Information Management System (LIMS). The collection of programs and procedures which comprise the LIMS is called "SAM." References made to SAM in this SOP are references to this collection of programs and procedures.

1.1.2 Sample entry must be performed in a timely fashion to allow tests with short holding times to be started immediately. Accuracy in the recording of sample IDs, in marking samples with lab numbers, and in checking for consistency of all records is of utmost importance.

1.2 Scope

1.2.1 All samples received by the laboratory are logged using the following procedures.

2. EQUIPMENT LIST

Lab coat
Disposable gloves
Respirator, dust mask
3M desk cleaner, broom, dustpan, mop
Spatula
Waterproof labeling gun
PC work station linked to SAM

3. SAFETY PRECAUTIONS

3.1 Sample Handling

3.1.1 Samples received at the laboratory can potentially be contaminated with toxic materials. Reasonable caution must be exercised at all times when handling these samples. Such precautions include wearing a lab coat at all times, using gloves, using a hood (located in Inorganics) to perform operations when necessary (strong odors present, etc.), and wearing a respirator or dust mask if fumes or dust are generated.

3.1.2 Cleanliness and neatness are of utmost importance. All spills and condensation from wet sample containers must be cleaned up immediately. This will help to alleviate accidental sample breakage and protect others from possible contact with contaminated work areas.

3.1.3 When wearing gloves, be certain to remove them when opening the cooler or lab doors and when answering the phone. The gloves which protect the sample enterer from contamination may transfer contamination to these objects. Other persons may touch the door knob or phone without glove protection and have the contamination transferred to their unprotected hands. Never put pens, paper clips, etc. in your mouth.

3.1.4 A dust mask is worn when pouring dry packing material such as vermiculite into the garbage.

4. OPERATION PROCEDURES

4.1 Sample Receipt

4.1.1 Samples may be received by client delivery, over the front counter, via UPS, courier services, by various air freight and overnight delivery services, and by Greyhound. It is the responsibility of the sample enterer to ensure that samples received by any of these services are promptly logged in and work requests made to the laboratory.

4.1.2 If a chain-of-custody (COC) is received with the sample set, sign it and record the date and time it was received. If the client has delivered the samples by hand, verify the cooler contents and return a copy of the COC to the client.

4.1.3 If complete verification of the cooler contents will occur later, then the COC is stamped and the stamped copy returned to the client. This stamp is reproduced in Appendix 1. Verification must take place within one working day of receipt.

4.1.4 All discrepancies between the COC and the actual samples received are immediately reported to the client and are noted on the Sample Receipt Log. CLP Sample Receipt Log (Appendix 3) is for CLP log-in procedure. **NON-CLP** Sample Receipt Log (Appendix 2) is for Laucks **NON-CLP** log-in procedure. If requested a client provided receipt form may be substituted for the Laucks sample receipt log.

4.1.5 Put on gloves, open the coolers (in the hood if necessary), and note whether custody seals are present and, if so, intact. **Affix one of the intact custody seals on the sample receipt log.** If there is a question about the integrity of the custody seals, make a note on the CLP Sample Receipt Log (Appendix 3); the client must be informed.

4.1.6 After the coolers are opened, determine whether there are soil or water samples in the coolers. Typically there will be a number of sample bottles for each sample if they are water; soils will have only a small number of containers per sample.

4.1.7 Visually check the contents of the opened cooler for obvious damage or broken sample containers. Note any breakage on the appropriate Sample Receipt Log.

4.1.8 For any program (such as HAZWRAP, NFESC, or Army Corps) or other project-related samples either the enclosed temperature blank or at least 3 separate containers taken randomly from different locations in EACH cooler must be checked for temperature with the infrared thermometer. The temperatures are recorded on the Supplemental Sample Receipt Log (Appendix 3). If any samples exceed the range of 4°C ± 2°C, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX) by the appropriate project manager. A copy of the communication from the Supplemental Sample Receipt Log must be kept with the COC in the work order file.

4.1.9 Remove all bottles from the cooler and put on the bench. Line up the bottles in some kind of order, if there is an apparent order. Various means of ordering samples are:

- COC order
- Client sample ID
- Date sampled
- Time sampled

4.1.10 For samples consisting of multiple containers, place all containers together on the bench.

4.1.11 After all samples are arranged then check consistency between the COC and the sample labels for Sample IDs, dates and times on each sample container.

4.1.12 Determine whether custody seals are present on the individual sample containers (jars and bottles). If present and intact, so note. If present and any seal is broken, so note. These notations must be made on the CLP Sample Receipt Log Form (Appendix 3).

4.1.13 All preserved water sample bottles for project-related work as well as unpreserved water sample bottles for HAZWRAP, NFESC, or Army Corps projects must also be checked for pH at the time of sample receipt. This is done by pouring out some of the sample into a small plastic cup and then using pH paper to record the pH at time of receipt. Volatiles samples should **NOT** be checked. When better discrimination of pH is needed, narrow range pH paper should be used to confirm the pH (especially if the pH is within 1 pH unit of the required preservation limit for that sample). All pH measurements must be recorded on the Supplemental Sample Receipt Log (Appendix 3). If any samples exceed the pH requirements, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX) by the appropriate project manager. The samples with inappropriate pH are listed on Laucks Testing Lab pH log form (Appendix 5) for corrective action. After the corrected preservation is completed this form is given to the appropriate project manager for work order filing.

4.1.14 Some samples are received at the lab that need to be split and preserved for different analytes. To accommodate preservation requirements, these samples are recorded on the "Sample Split Sheet" (see Appendix 6). There is a specific cart located in sample entry where the samples are temporarily stored until splitting and preservation take place.

4.1.15 All sample container marks (including ID's, dates and times) are then verified with each other and with the COC. This is done by noting whether all bottles from the same sample have the same ID and whether this ID is the same as on the COC. All discrepancies are noted on the Non-CLP Sample Receipt Log (Appendix 2) or the CLP Sample Receipt Log (Appendix 3) and reported to the client.

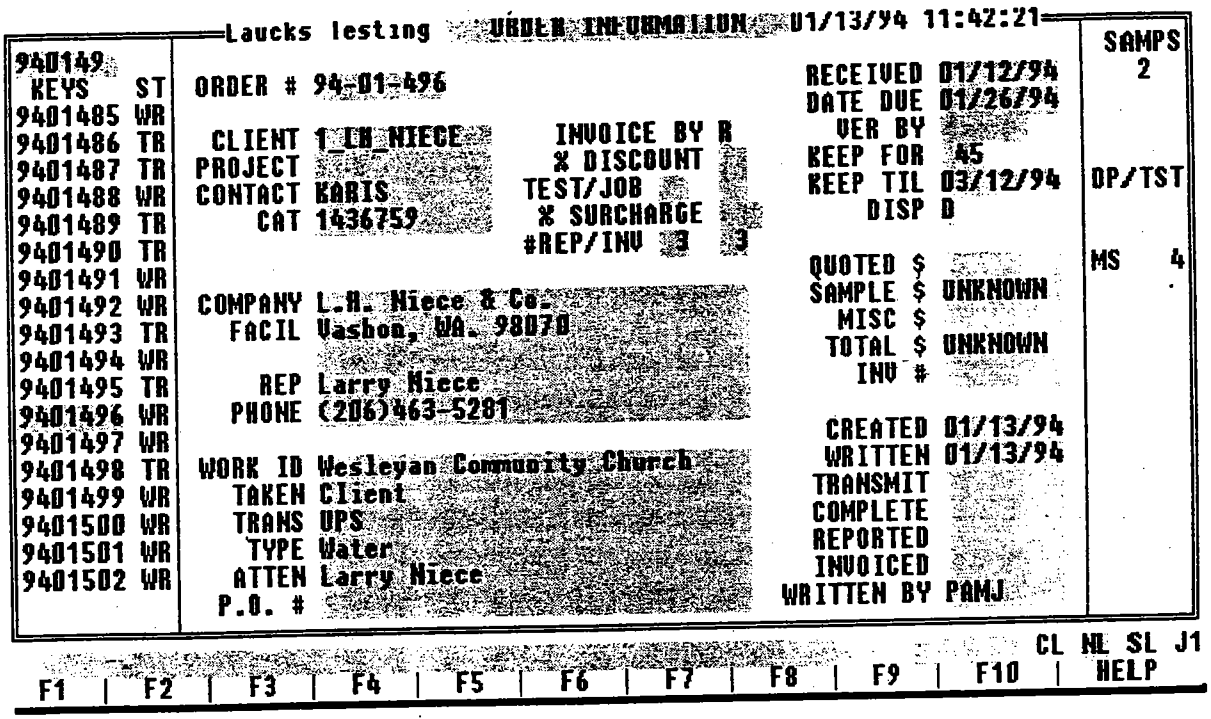
4.1.16 To determine if the sample(s) is(are) acceptable, compare the existing conditions with the criteria specified in Appendix 7, "Required Containers/Volumes, Preservation Techniques and Maximum Holding Times for Environmental Analysis". All listed criteria must be met in order to qualify the sample(s) as "acceptable". If there are any problems with the sample(s) these must be documented in the "CLP Sample Receipt Log" (see Appendix 3). If any samples are not acceptable, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX) by the appropriate project manager.

4.2 Sample Log-In

4.2.1 Determine whether a client record exists in the SAM database. If it does not, create a record. At a minimum, the client record will include:

- an alphanumeric client code (up to 12 digits)
- the client's full and accurate name, address, and point of contact
- the client's telephone number and/or FAX number
- the full and complete address for invoices
- the purchase order/contract number <u>if</u> that number applies to <u>all</u> work the client may submit. (If the purchase order/contract number is specific to one sample submittal, by project etc.), then the client code would be project specific. Example (client name_project name).

4.2.2 A SAM work order is started for the job through the ORD program. The work order is identified by a unique 7-digit number which is assigned by SAM at the time the work order is initiated. (The first two digits of this number represent the year, the third and fourth digits represent the month, and the final three digits represent the work order's sequence within the month. For instance, work order 9004001 was initiated in April, 1990 and was the first work order for that month.) This number will be used throughout the laboratory to track the job.



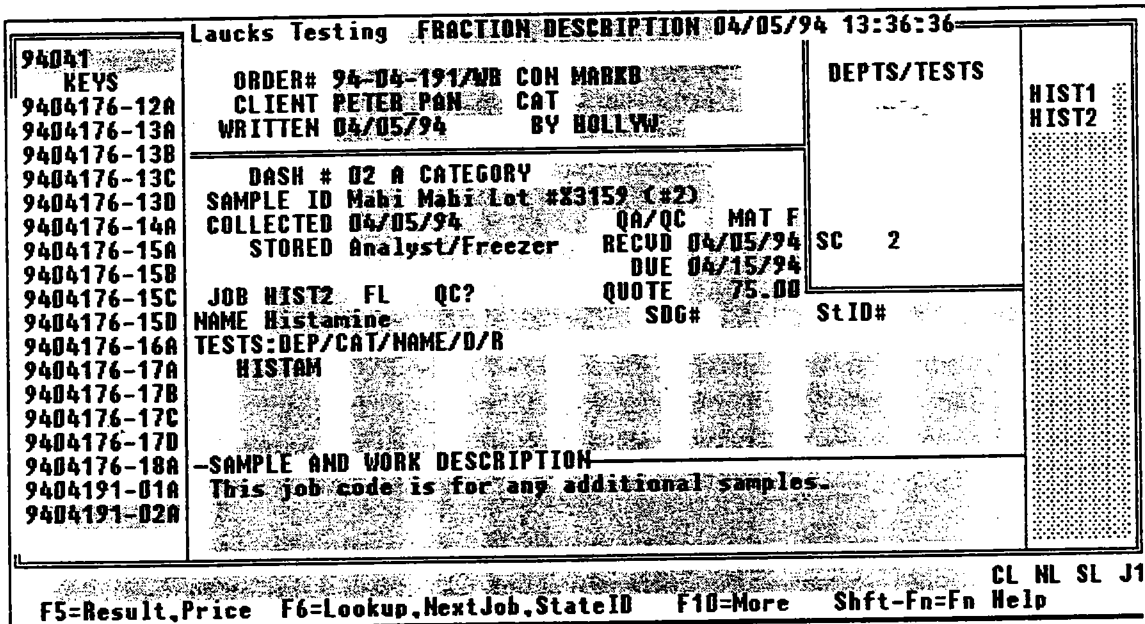
ORD Screen

4.2.3 The work order is to be filled out as completely as possible at this time. Above is an example of what the work order screen looks like on your PC. Typical information put into the work order screen (analogous to a cover page) includes:

- date of sample receipt;
- work order due date;
- client point of contact (if different than in the client record);
- sample type (soil, water, etc.);
- the manner in which the samples were received at the laboratory (hand-delivered, Greyhound, etc.);
- air bill number (or equivalent) if the sample was transported by common carrier;
- the client's overall project identification (both the name of the project and any project, job, or purchase order number);
- and any relevant surcharges or discounts to be applied at the time of invoice.

4.2.4 All required data entry fields are in inverse video (highlighted) on the screen, but fields for purchase order numbers, project name or number, and point of contact should also be completed, if the information is known.

4.2.5 All sample IDs, dates of collection, and dates of receipt are recorded in the FRAC program for the work order with which they are associated. If there is a discrepancy in identification between bottles of the same sample, make a note on the appropriate sample receipt log and the project manager will notify the client.



FRAC Screen

4.2.6 All sample bottles are numbered with the work order number and a fraction (or sample) number. Fraction numbers are assigned sequentially to each sample based on the order in which the samples were sorted and logged (COC order, client sample number order, etc.). This number is used to track the sample throughout the laboratory. See section 4.2.10 for specifications for unique bottle identifiers required by Navy projects.

4.2.7 A sample can be uniquely identified by its work order number and the fraction number. For instance, if the work order number was 9004001 and there were 4 samples, the job would consist of samples

9004001-01
9004001-02
9004001-03
9004001-04

4.2.8 Each sample or set of samples is assigned a unique identifying work order number, generated by the Laboratory Information Management System (LIMS), on receipt. This unique number consists of 7 numerical characters, such as 9007215. In this example, the work order was initiated in 1990 (90), in the month of July (07) and was the 215th such work order that month (215). If more than 999 workorders are generated in any month, the 5th digit is replaced by successive letters of the alphabet (A-Z).

4.2.9 If necessary, more than one fraction may be created for a sample (generally, this is related to billing issues - when one analysis is discounted in price and another is not, for instance), but they will all bear the same fraction number and be differentiated by an automatically-assigned letter suffix. For instance, if sample 9004001-01 had 3 fractions, purely for internal accounting reasons, the three fractions would be identified as:

9004001-01A
9004001-01B
9004001-01C

4.2.10 The person performing log-in needs to be aware of this effect, but it has no impact on sample identification within the lab, on sample tracking, or on the sample number placed on the bottles/jars. In the above example, all bottles submitted for this sample would be marked 9004001-01.

4.2.11 For Navy projects, each bottle must have a unique bottle identifier. Every bottle must have a specific 1-3 digit numerical identifier that is unique to each bottle submitted within a workorder. The numbers are assigned in consecutive order so that all bottles of similar size/type with the same preservation for the same analysis (analyses) from a particular workorder will have consecutive bottle numbers. The first bottle of each analysis type in each new workorder starts over again with bottle number 1.

4.2.12 This information must be recorded in the "Bottles" computer tracking program under each workorder and the workorder-unique bottle identifier will be printed in the bottom left corner of each bottle label (which also contains the workorder number and the sample number) before the bottle label is affixed to each individual bottle.

4.2.13 **ALERT:** Each SAM work order can accommodate up to 57 fractions and no more. Each work order must allow sufficient fraction space for later changes or additions. Therefore, no more than 50 samples should be logged into any single work order. If, for administrative reasons, some or all of the samples consist of more than one fraction, then no more than 50 fractions can be logged. Should the submittal consist of more than 50 samples, or more than 50 fractions, initiate additional work orders as required. Cross-reference the work order numbers, so that all samples submitted together can be reported together to the client. You can perform this cross-reference manually (by noting on accompanying documents "See Also [Work Order Number]) or you can make appropriate comments in the Work Order Comment field (F2). To the degree possible, make sure that multiple work orders which represent one complete project in the client's mind are created sequentially, with no other unrelated work order numbers intervening.

4.2.14 Additionally, Sample **D**elivery **G**roups (SDGs) are commonly created for project work at the time of sample entry. SDGs consist of no more than 20 samples being analyzed for the **same** test. This is in order that the appropriate amount of QC may be analyzed and reported with any sample set. Specifics of the SDG creation process are outlined elsewhere in this SOP.

4.2.15 **ALERT:** Each fraction will accommodate only 27 tests. If more than 27 analyses are required on any sample, additional fractions should be made (i.e. -1A, -1B, -1C, etc.).

4.2.16 **ALERT:** The work order will accommodate only one date of receipt, while each fraction will accommodate individual receipt dates and due dates. If samples are submitted over several days, and are logged into one work order, the Sample Custodian **MUST** enter appropriate dates of receipt in each fraction. The FRAC program will default to the current date. If the samples were received on an earlier date, that date **MUST** be entered for that fraction for the date to be correct. Similarly, the fraction due date will default to that of the workorder on the ORD screen. If different fractions of the same sample are due at different times, due to client or other demands, the date they are due **MUST** be entered for that fraction.

4.3 Special documentation procedures for CLP samples

4.3.1 Completion of the CLP Sample Receipt Log, and the Supplemental Sample Receipt Log

4.3.2 CLP Sample Receipt Log and the Supplemental Sample Receipt Log are CLP-specific sample login sheets. For each cooler received a CLP Sample Receipt Log Form and a Supplemental Sample Receipt Log must be completed. This form takes the place of the NON-CLP Sample Receipt Log (Appendix 2). Copies of these forms may be found in Appendix 3.

4.3.3 Complete the header information requested at the top of the forms. Use multiple pages if necessary.

- date received
- time received
- client name
- SDG #
- COC # (if available)
- sample log-in date
- work order #
- client project
- airbill number (if available)
- and initials of the person logging in the samples.

4.3.4 Complete the Non-Conformance check list. If there is a problem with the custody seals, chain of custody records, or agreement between the custody records, the client must be contacted. In this case, this should be done **in writing** (preferably FAX) by the appropriate project manager. A copy of the communication must be kept with the COC in the work order file.

4.3.5 Since the extractable fractions will be transferred to the extractions lab, a Secure Storage Custody Log must be completed, and the samples are held on 8C in the WO1 walk-in cooler (extractions hold shelf) pending pick-up by extractions personnel. Specifics of the Storage Custody Log is outlined in the Chain-Of-Custody SOP located in the SOP manual.

4.4 Assignment of SDG numbers

4.4.1 The SDG name is assigned by sample control and is usually based on client name or project name followed by sequential numbering.

4.5 Assignment of lab quality control samples

4.5.1 The client may choose to designate which samples are to be analyzed as matrix spike/matrix spike duplicate samples. This means that the sample preparations and the VOA departments cannot self-assign QC samples until all samples from the SDG are received. It is the responsibility of the sample login person to notify the operations staff when a specified QC sample is received.

4.5.2 Note in the **SAM SDG** records which sample is QC-assigned.

4.5.2.1 This information is pulled into SDG from the FRAC program when the SDG is created.

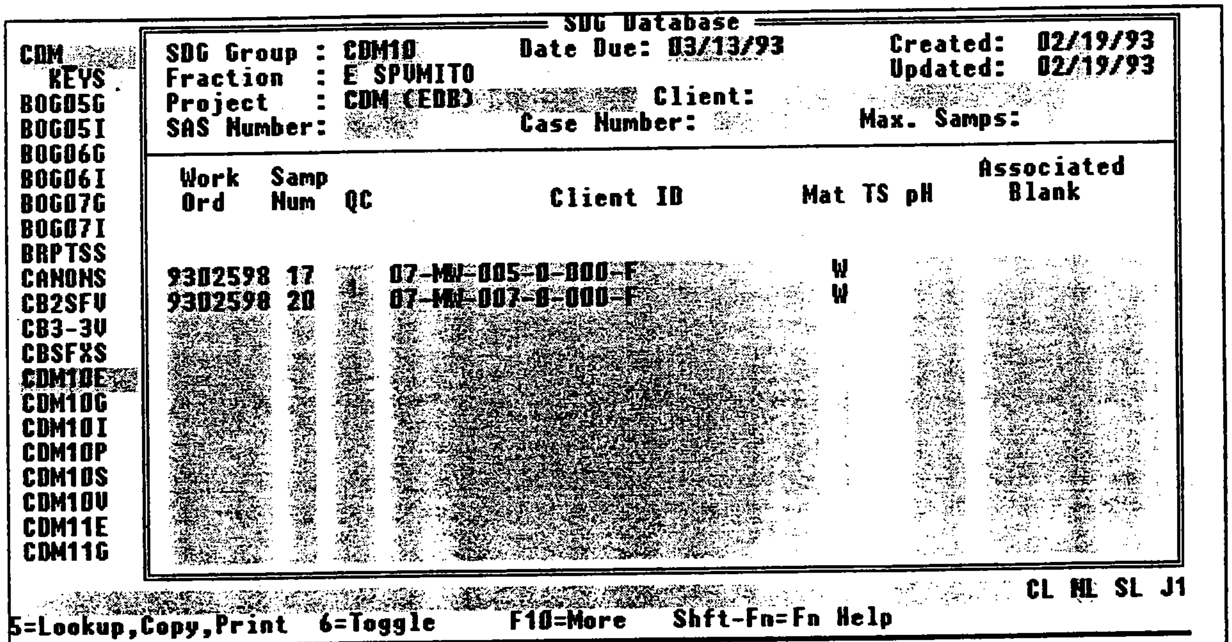
4.5.2.2 On the FRACTION screen, an X is placed in the QC field. This will print out next to the sample and indicate that it is an assigned QC sample.

4.5.2.3 Write a department comments message for whichever department (EX, MS, and/or GC) should be notified.

4.6 Completion of SDG records.

4.6.1 For each project (NFESC, HAZWRAP, or Army Corps) SDG records in SAM are created. The SDG concept follows the CLP model: that is, up to 20 samples of similar matrix and analytical fraction are grouped together for preparation and analysis. Samples are assigned to SDGs at sample login and are also reported by SDG.

4.6.2 The **SDG** program is an electronic means of compiling information about the samples assigned to an SDG in one place. Example **SDG** screens are reproduced on the following page:



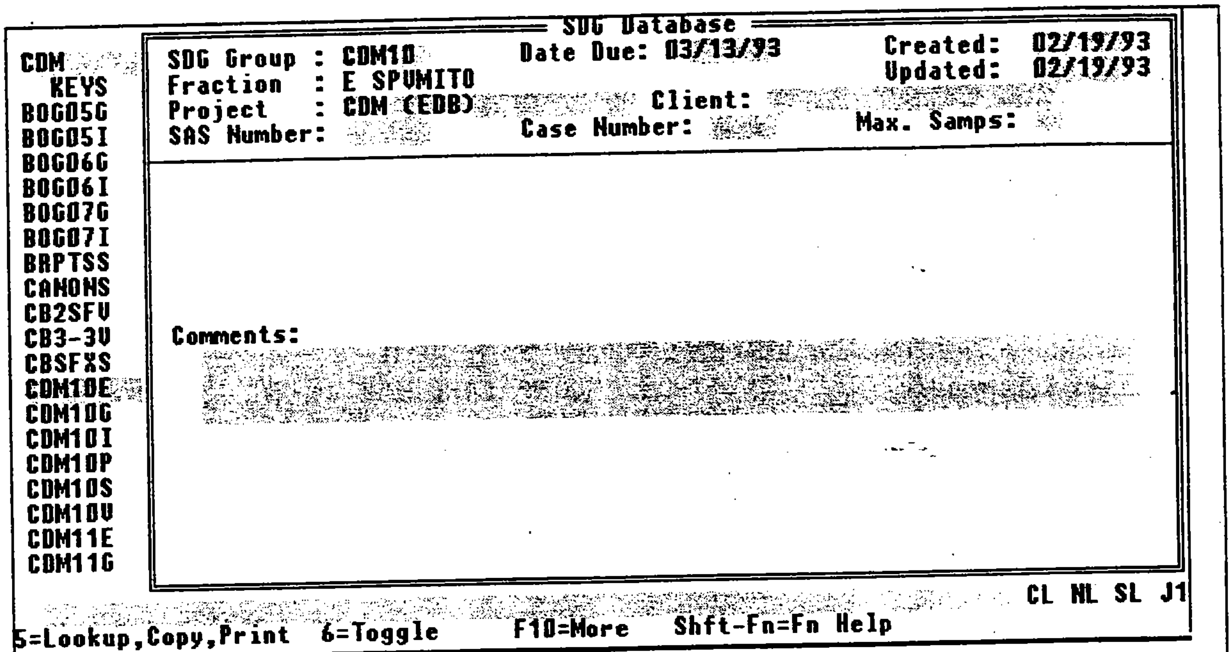
SDG, Screen 1

```
                         SDG Database
CDM [redacted]  SDG Group  : CDM10[redacted]   Date Due: 03/13/93        Created:  02/19/93
 KEYS           Fraction   : E SPUMITO                               Updated:  02/19/93
BOG05G          Project    : CDM (EDB)[redacted]      Client: [redacted]
BOG05I          SAS Number: [redacted]   Case Number: [redacted]   Max. Samps: [redacted]
BOG06G
BOG06I           Work   Samp              Date
BOG07G           Ord    Num    UTSR       Collected
BOG07I
BRPTSS
CANONS          9302598 17  02/16/93  02/13/93
CB2SFU          9302598 20  02/16/93  02/13/93
CB3-3U          [redacted]
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10U
CDM11E
CDM11G
[redacted]                                                        CL  NL  SL  J1
5=Lookup,Copy,Print   6=Toggle      F10=More    Shft-Fn=Fn Help
```

SDG, Screen 2

```
                         SDG Database
CDM [redacted]  SDG Group  : CDM10[redacted]   Date Due: 03/13/93        Created:  02/19/93
 KEYS           Fraction   : E SPUMITO                               Updated:  02/19/93
BOG05G          Project    : CDM (EDB)[redacted]      Client: [redacted]
BOG05I          SAS Number: [redacted]   Case Number: [redacted]   Max. Samps: [redacted]
BOG06G
BOG06I           Work   Samp                     Fractions
BOG07G           Ord    Num    EDB  [redacted]
BOG07I
BRPTSS
CANONS          9302598 17     X
CB2SFU          9302598 20     X
CB3-3U          [redacted]
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10U
CDM11E
CDM11G
[redacted]                                                        CL  NL  SL  J1
5=Lookup,Copy,Print   6=Toggle      F10=More    Shft-Fn=Fn Help
```

SDG, Screen 3



```
                    SDG Database
CDM      SDG Group  : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction   : E SPUMITO                              Updated:  02/19/93
BOG05G   Project    : CDM (EDB)               Client:
BOG05I   SAS Number:               Case Number:              Max. Samps:
BOG06G
BOG06I
BOG07G
BOG07I
BRPTSS
CANONS
CB2SFU
CB3-3U   Comments:
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10U
CDM11E
CDM11G
                                                             CL  NL  SL  J1
5=Lookup,Copy,Print   6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 4

4.6.3 Fill in the header section of the first screen page. For CLP cases, fill in the Fraction (V=Volatiles, P=Pesticides, S=Semivolatiles etc..) Indicate the project name, and the client's name.

4.6.4 When the work order number and sample number are entered, the sample-specific information shown in screens 1, 2, and 3 is read in from the SAM database. (**Hint**: after the first work order number is entered, it is only necessary to enter sample numbers for subsequent samples from the same work order.)

4.6.5 On screen 3, a table of fractions/tests is created. An 'X' is entered to signify that a particular test is required on a given sample.

4.6.6 Each 'fraction' has a separate SDG entry. For instance, VOAs and ABNs are entered on separate SDG records, as indicated above (V=Volatiles, S=Semivolatiles, P=Pesticides). A single letter suffix (V, S, P etc...) is assigned to each SDG record before it is saved to disk. The end result is that you may have multiple SDG records for a given SDG, each with the same root name, but a different suffix. This system is used to allow for the possibility that within the same SDG, varying numbers of tests will be assigned to samples within that SDG.

4.6.7 The last screen page is used for any comments which the sample login person or project manager would like to record for the operations staff.

4.7 Sample storage

4.7.1 The following tests must be started very soon after receipt when performed on water samples.

Test Type/Name	SAM Code(s)
NO_3 - nitrate	NO3ICW
NO_2 - nitrite	NO2_W, NO2_DW
ortho phosphate and soluble reactive phosphate	P04O_W, P04S_W
Cr^{+6} - hexavalent chromium	CR6_W, CR6_WM
CO_2 - carbon dioxide	CO2_N
DO - dissolved oxygen	DO_W
BOD - Biological Oxygen Demand	BOD_5
Chlor A - Chlorophyll A	CHLORA
Settleable Solids	SETSOL, SETSL2
Filtration for dissolved metals	FILTER
pH	PH_EPW, PH_SWW
Microbiological tests	[various]
Color	COL_DW
Turbidity	TUR_TW, TUR_W
Sulfite	SO3_W
MBAS - Methylene Blue Active Substances	MBAS
Chlorine	CL2_R

4.7.2 A rush backlog report is printed throughout the day for short holding-time tests, with the exception of microbiology, in order that they be recognized by the analysts. **A checklist (Appendix 8) for analyses with short holding times is completed prior to release of samples to the laboratory. Sample management will verify that the correct RUSH test codes are entered, date, time received and collected dates are accurate and to ensure matrices and sample I.D.s are correct at time of log-in.**

4.7.3 For microbiological samples and for samples which arrive late in the day and for which the holding time will expire if the analysis is not started that day, the containers must be taken immediately to the work areas in which the tests will be performed and the primary person responsible for these tests notified that samples are here. A list with the name of the appropriate analyst is posted in the sample entering area.

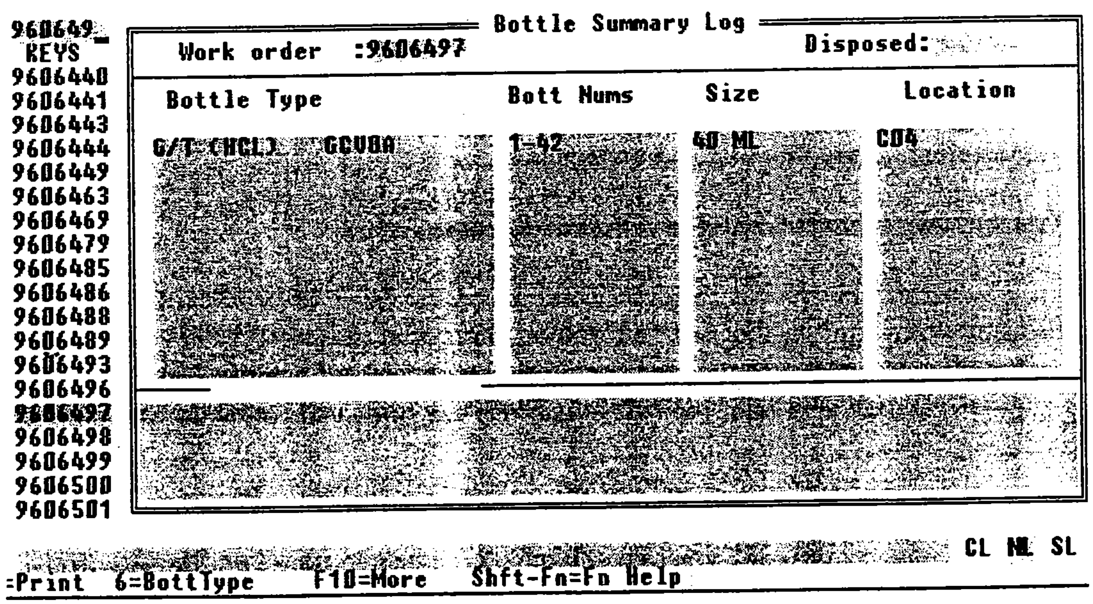
4.8 Storage locations

Location	Description of contents
VOA refrigerator	Soil and water volatiles
Shelf	Aqueous metals, oils, no cooling required
Inorganics cooler	Complete small water jobs
Walk-in cooler	All other soil and water*
Section in the walk-in cooler indicated w/CLP sign-in, sign-out sheets	All samples that are under internal COC. CLP samples are also stored in here.

4.8.1 *See Section 4.7 for transfer of extractable aliquots to the extractions lab

4.8.2 Prior to putting bottles into any storage location, the electronic Bottle Summary Log must be completed. For Navy projects, unique bottle identifiers must also be entered in the bottle log.

4.8.3 At the J1 prompt, type "BOTTLES." This log details how many bottles were received, what type and size of bottles were received, the storage location of the bottles and the bottle numbers, where applicable. An example of this log follows.



4.9 Determination of tests

4.9.1 If Laucks provided sample bottles for the client, the bottle order, the client COC, file notes, letters, client instructions, or the client file are consulted as necessary to determine what tests are to be performed. The type of bottles received for water samples will help greatly in determining which tests to perform. If you can't determine the tests, give the paperwork to the Project Manager, who will contact the client.

4.9.2 A lab work request is initiated at this time. Based on a review of the above information, test codes are assigned to the appropriate fractions. These test codes may represent single-data point analyses ("regular" tests) or multiple-data point analyses ("special" tests), such as GC/MS volatiles. However, no work request packet can be prepared until after "transmittal," which is initiated by the Project Manager or designee.

4.9.3 Some soil samples will need to be shared between two or more departments. In order of priority, the following areas will receive samples in this order:

4.9.4 If volatiles are requested, then the VOA departments will get the samples first (GC or GC/MS)

4.9.5 The sample/samples will then be sent to the extractions lab

4.9.6 The extractions lab will return the sample/samples to the inorganics lab or other areas

4.9.7 At the time of sample log-in the Sample Custodian will make appropriate comments for the department to return the samples to other departments for further testing. **Before** any samples are sent to other departments for testing, it is imperative that any requiring the analysis of volatile organics gets the sample first. Such samples should be given to the Volatiles Department before any other department.

4.10 Electronic Transmittal of Sample and Test Request Records

4.10.1 Specifics of the transmittal process are detailed in a separate SOP. A brief summary follows. For actual transmittal, that SOP should be referenced as it will detail greater specifics and will contain changes that may occur in the transmittal process. The following is only intended as a brief overview and may not reflect the most current practices.

4.10.2 All documentation (including, but not limited to, air bills, chain-of-custody documents, bottle order forms, notes, contracts, messages, letters, etc.) that supports the information entered into the work order and the sample fractions is clipped together by the Sample Custodian when sample log-in is complete. The work order number is written on, at a minimum, the chain-of-custody document and may also be written on any other relevant documents.

4.10.3 The supporting documentation is given to the project manager, her designee, or to the head of the Project Management Group for "transmittal." Transmittal is the electronic approval of the work order and sample fractions as written and must be performed within 1 working day of sample log-in. Transmittal is the activity which electronically puts the samples and test requests into the laboratory's analytical schedule.

4.10.4 In performing the transmittal, it is the responsibility of the Project Manager, or designee, to double-check the work order and test fractions for the following:

- accuracy of project information (number, name, point of contact, etc.)
- accuracy of test requests
- and accuracy of the test codes employed to represent those test requests.

4.10.5 The Project Manager makes corrections to these items as necessary, usually in consultation with the Sample Custodian. When transmittal is complete, the hard-copy record generated in the transmittal process is stapled to the supporting documentation previously assembled by the Sample Custodian and the complete record is filed alphabetically (by client name) in the filing drawer designated. If a CLP-style package is being generated, packets are prepared for the CLP Document Control Custodian.

4.10.6 Specific test requests are made known to analysts through hard-copy work "backlogs". For a description of this process, see the SOP on Data Handling.

4.11 Generation of internal Chain-of-Custody (COC)

4.11.1 Samples which must be removed from the main building at 940 and taken to the extractions lab at 921 for preparation are tracked with an internal COC. This form is initiated by the person logging in the samples. The lab number, the client name, the number of samples, the sample matrixes and the enterer's initials and the date and time the form is started are recorded. See Appendix 4 for an example of an internal COC.

4.11.2 The samples are placed on shelf 8C in the walk-in with the COC. The person removing the samples from 940 signs and dates the form. The samples are logged into a log book at 921 before being placed in the cooler.

4.11.3 The COC is returned to 940 with the extracts when extractions are completed.

4.12 Sample breakage

4.12.1 All sample breakage, whether in shipping or while handling in the lab, must be reported immediately to the Project Manager.

4.12.2 If the sample was water, clean thoroughly with disposable towels. Be very careful with broken glass so as to avoid cuts.

4.12.3 If the sample was soil, as much of the sample as possible is transferred to a new, clean jar using a spatula. Be certain not to pick up any sample which has contacted the floor. Save the original label, if possible. Note on the log-in records that the sample was broken and transferred to a new container.

4.12.4 All dirty, disposable clean up materials, soil, broken glass, etc. are placed in a plastic garbage bag before being placed in the dumpster. Any non-disposable clean up materials are washed after use.

4.13 Special circumstances

4.13.1 Samples from some clients are logged into a monthly work order. Some jobs extending over more than one sampling event may be entered under one work order number. In that event, pay special attention to date of receipt (see ALERT, above).

4.13.2 Other special circumstances may arise. If there are any questions, check with the Project Manager first.

APPENDIX 1

Sample Receipt Verification Stamp

Sample Receipt acknowledged pending verification of sample count. You will be notified within one working day of any discrepancies found

Signed ________ Date ______ Time ______

APPENDIX 2

NON-CLP Sample Receipt Log

Laucks Testing Laboratories, Inc.
NON-CLP SAMPLE RECEIPT LOG

Initial once samples are checked in__________

DATE RECEIVED:____________
TIME RECEIVED:____________
CLIENT NAME:____________
SDG #____________
COC #____________

SAMPLE LOG-IN DATE:____________
WORKORDER #:____________
CLIENT PROJECT:____________
AIRBILL ATTACHED?:(#)____________
RECEIVED BY:____________

Non-Conformance: (Check applicable item(s)) Client IDs affected:

°(1) Not enough sample sent for proper analysis. #s affected:____________
☐(2) Sample Bottle received broken and/or cap not intact.____________
☐(3) Custody seal: Absent____ Present/Intact_____ Present/Broken ________
☐(4) Any temperature out of compliance:____________
☐(5) Sample received outside of holding time.____________
☐(6) Sample not properly preserved. pH = ___. Wrong preservative used.____________
☐(7) Illegible sample numbers or label missing from bottles.____________
☐(8) Identification on bottle same as identification on paperwork: yes:_____no:________
☐(9) Incomplete instructions received with sample(s), i.e.,
☐ no Request for Analysis. no Chain-of-Custody.____________
☐(10) Samples received in improper container.____________
☐(11) Samples held in field before receipt by Lab. Days (specify)____________
☐(12) Air Bubble(s) in ___of ____ samples for volatiles analysis.____________
☐(13) Other ____________

CORRECTIVE ACTION: (Check applicable item(s)
Correction action taken by:

	Inititals	Date
☐(1) Client informed verbally (Client Services).	______	
☐(2) Client informed by memo/letter/fax (Client Services).	______	____
☐(3) Sample processed "as received" (Sample Entry).	______	____
☐(4) Re-sampling requested of client (Client Services).	______	____
☐(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	______	____
☐(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	______	____
☐(7) Other (Specify)____________	______	____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

APPENDIX 3

CLP Sample Receipt Log

Supplemental Sample Receipt Log

COOLER RECEIPT FORM

WORKORDER #: ________________

Contractor Cooler ________________
QA Lab Cooler # ________________
Number of Coolers ________________

Project: ____

Date samples were received at the laboratory: ___/___/___

A. **PRELIMINARY EXAMINATION PHASE:** Date cooler was opened: ___/___/___

By: (print)__________________________(sign)__________________________

1. Did cooler come with a shipping slip,(airbill, etc.),>>>>>>>>>>>>>>>>>>>>>> YES NO
   If YES, record carrier name and airbill number: ____________________________
2. Were custody seals on outside of cooler?>>>>>>>>>>>>>>>>>>>>>>>>>>> YES NO
   How many and where: ____
   Seal date: ___/___/___Seal name: ________________
3. Were custody seals unbroken and intact at the date and time of arrival?>>>>>> YES NO
4. Did you screen samples for radioactivity using the Geiger Counter?>>>>>>>> YES NO
5. Were custody papers sealed in a plastic bag and taped inside to the lid?>>>>>> YES NO
6. Were custody papers filled out properly (ink, signed, etc.)?>>>>>>>>>>>>>> YES NO
7. Did you sign custody papers in the appropriate place?>>>>>>>>>>>>>>>>>YES NO
8. Was project identifiable from custody papers? If YES, enter project name at top of this form.
9. If required, was enough cooling material present?>>>>Type of ice: ________YES NO
10. Have designated person initial here to acknowledge receipt of cooler: ____date___/___/___

B. **LOG-IN PHASE :** Date samples were logged-in: ___/___/___

By (print)__________________________(sign)__________________________

11. Describe type of packing in cooler: ____________________________________
12. Were all bottles sealed in separate plastic bags?>>>>>>>>>>>>>>>>>>>>>> YES NO
13. Did all bottles arrive unbroken and were labels in good condition?>>>>>>>>> YES NO

Page 1 of 3

LAUCKS TESTING LABORATORIES, INC.
COOLER RECEIPT FORM (continued)

14. Were all bottle labels complete (ID, date, time signature, preservative, etc.)? YES NO
15. Did all bottle labels agree with custody papers?>>>>>>>>>>>>>>>>>>>>>>>YES NO
16. Were correct containers used for the tests indicated?>>>>>>>>>>>>>>>>>>>YES NO
17. Were correct preservatives added to samples?>>>>>>>>>>>>>>>>>>>>>>>>YES NO
18. Was a sufficient amount of sample sent for tests indicated?>>>>>>>>>>>>>> YES NO
19. Were bubbles absent in VOA samples: If NO, list by QA #:>>>>>>>>>>>>> YES NO
20. Was project manager called / faxed & status discussed? >>>>>>>>>>>>>>> YES NO
    If YES, give details below
21. Who was called / faxed? ______________________________
    By whom?______________________(date)______________

DISCREPANCIES:

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____

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

Work Order Number:________________

Assigned SDG Number:______________

Temperature		pH of Bottle Types								

Allowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

Temperature	Allowable temperature range is 4± 2 degrees Celsius
Acid Preserved pH	pH must be less than 2
Base Preserved pH	pH must be greater than 12

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APPENDIX 4

Secure Storage Custody Log

Organic Extractions Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:____________________ **LTL Number:**____________________

Number of Containers (*optional*):____________________

Storage Unit:____________________ **SDG Number** (*optional*):____________________

Sample Numbers	Matrix (*optional*)	Location (*shelf*)	Logged Out Date	Logged Out Time	By	Logged In Date	Logged In Time	By	Action

Samples Disposed of by ____________________ on ____________________

Laucks Testing Laboratories, Inc.

Organic Extractions Custondy Log

Samples Entered By	Time	Date	JOB #	
			Client	
Samples Moved From Bldg. 940 to 921 by:			*Matrix*	*Sample #*
			SOIL	
			WATER	
			SLUDGE	
			MISC.	
			OIL	

Comments:

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APPENDIX 5

Laucks Testing Lab pH Log Form

Laucks Testing Lab pH log Form

Date____________

Analyst	WO Number	Fractions	pH before	Volume Acid Added/Type	Resulting pH

APPENDIX 6

Laucks Testing Lab Sample Split Sheet

Laucks Testing Lab Sample Split Sheet

WO #	Sample #	WE HAVE	WE NEED	TOTAL	DISS	UNPRES	INTL & DATE	BOTTLE TYPES
								Y = YELLOW
								B = BLUE
								C = CYANIDE
								R = RED
								O = O&G
								P = PHENOL
								S = SULFIDE
								T = TOC
								OTHER

APPENDIX 7

Required Containers/Volumes/Preservation/Holding Times

REQUIRED CONTAINERS/VOLUMES, PRESERVATION TECHNIQUES AND MAXIMUM HOLDING TIMES FOR ENVIRONMENTAL ANALYSIS

A. PRIORITY POLLUTANTS - Organics Analysis (Federal Register Vol. 49, No. 209, October, 1984)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Purgeable Halocarbons	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, no headspace	14 days, with preservation	Method 601, GC/ELCD or Method 624, GC/MS
Purgeable Aromatic Hydrocarbons	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, adjust pH to $\leq$ 2 HCl, no headspace	14 days, with preservation 7 days, if not preserved	Method 602, GC/PID or Method 624, GC/MS
Acrolein and Acrylonitrile	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid, no headspace As above, and pH adjusted to pH 4-5.	7 days 14 days	Method 624, GC/MS
Phenols	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.008% $Na_2S_2O_3$ pH <2 H_2SO_4.	7 days until extraction; 40 days after extraction for analysis	Method 625, GC/MS

A. PRIORITY POLLUTANTS - Organics Analysis (Federal Register Vol. 49, No. 209, October, 1984) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Pesticides (Organochlorine Pesticides, and PCB's	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 608, GC
Polynuclear Aromatic Hydrocarbons (PAHs)	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 610, GC or Method 625, GC/MS
Base/Neutral and Acid Extractables	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 625, GC/MS

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Halogenated Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, no headspace Cool, 4° C, packed to avoid headspace	14 days 14 days	Method 8010 GC/Hall - Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030
Nonhalogenated Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH ≤ 2 with HCl, no headspace Cool, 4° C, packed to avoid headspace	14 days, with preservation 7 days, if not preserved 14 days	Method 8015 GC/FID Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Aromatic Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid and adjust pH to $\leq$2 with HCl, no headspace Cool, 4° C	14 days with preservation 14 days	Method 8020, GC/PID Direct Injection or Headspace, Method 5020 - Purge-and-Trap, Method 5030 Method 8260/8240, GC/MS Purge-and-Trap Method
Acrolein, Acrylonitrile Acetonitrile	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH 4-5 with HCl, no headspace Cool 4° C, no headspace	14 days, with preservation 14 days	Method 8030, GC/FID Direct Injection or Headspace, Method 5020 - Purge-and-Trap Method 5030 - Groundwater using Method 5030 only. Method 8260/8240, GC/MS Purge-and-Trap Method
Phenols	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C, 35 mg $Na_2S_2O_3$ per ppm free chlorine per liter, adjust pH <2 with H_2SO_4 Cool 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8040 GC/FID or GC/ECD or Method 8270 GC/MS
Organochlorine Pesticides and PCBs	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C, adjust pH to 6-8 with H_2SO_4 or NaOH Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8080 GC/ECD

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Polynuclear Aromatic Hydrocarbons (PAHs)	Approximately 1 liter for liquid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days	Method 8310 HPLC or
	Approximately 50 grams for sludge or solid sample			Extracted within 14 days and completely analyzed within 40 days	Method 8270 GC/MS
Chlorinated Hydrocarbons	Approximately 1 liter for liquid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days	Method 8270 GC/MS
	Approximately 50 grams for sludge or solid sample			Extracted within 14 days and completely analyzed within 40 days	
Organo-phosphorus Pesticides	Approximately 1 liter for liquid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days	Method 8140 GC/NPD or GC/NPD/ECD
	Approximately 50 grams for sludge or solid sample			14 days	
Chlorinated Herbicides (i.e., 2,4-D and 2,4,5-TP)	Approximately 1 liter for liquid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days	Method 8150 Extraction and Esterification/GC-ECD
	Approximately 50 grams for sludge or solid sample			Extracted within 14 days and completely analyzed within 40 days	

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Volatile Organics (VOAs)	2- 40 ml containers for liquid sample 20 grams for solids	Glass, Teflon-lined Septum 40 ml capacity As above or glass, 2-4 oz.	Cool, 4° C, acid preserved with HCl to pH < 2, no headspace Cool, 4° C, no headspace or if solid packed to minimize headspace	14 days 14 days	Method 8260 Purge-and-Trap GC/MS
Semi-Volatile Organics	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C,	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8270 GC/MS

C. WASTE EVALUATION - GENERAL

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Toxicity Characteristic Leaching Procedure	Approximately 1 liter for liquid sample Approximately 200 grams for solid sample	Glass, Teflon-lined cap	VOA, Metals, Semivolatiles, Pesticides/Herbicides - 14 days until extraction. Follow analytical protocol for aqueous holding time or holding time from leachate preparation.	Not specified	According to requested analysis
Water Reactivity	Approximately 100 ml for liquid sample Approximately 50 grams for solid sample	-----	None	14 day	-----

C. WASTE EVALUATION - GENERAL (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Ignitability	Approximately 200 ml or 50 grams	None specified	None	14 days	Method 1010 Pensky-Martens Closed-Cup Method Method 1020 Setaflash Closed Cup Method
Corrosivity	100 - 500 ml	Plastic	None	None	Method 1110 Corrosivity Toward Steel
California Assessment Manual CAM/STLC	Approximately 200 ml for liquid sample Approximately 10 grams for solid sample	Plastic or Glass	No preservation for solid sample. Add HNO_3 to pH <2 for liquid sample	28 days for Mercury 6 months for others	See Methods for Metals Analysis

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition)

PARAMETER	MINIMUM VOLUME REQUIRED **	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Mercury, Total/Dissolved Mercury, Total	100-200 ml for liquid sample Approximately 5 grams for solid sample	Plastic or Glass	HNO_3 to pH <2 for total Filter on site, HNO_3 to pH <2 for dissolved	28 days	EPA 245.1 for water or EPA 7470A EPA 7471A for sediment Cold Vapor Method
Metals, Total Metals, Dissolved	300 ml for liquid samples Approximately 10 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 for total Filter on site, HNO_3 to pH <2 for dissolved	6 months	Flame AA-- See Individual Metal Methods Emission AA-- See Individual Metal Methods Graphite Furnace AA- See individual Metal Methods or ICP-- 200.7 or 6010

** For individual metals the aggregate minimum volume is determined by the number of discrete analytical methods not the sum of all the individual analytes.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Hexavalent Chromium -Cr[6]	200 ml for liquid sample Approximately 50 grams for solid samples	Plastic or Glass	Cool, 4 C	24 hours Extracted within 7 days, analyzed within 24 hrs. of extraction.	EPA 218.4 or EPA 7196A EPA 218.5 or EPA 7197 Extraction/AA Method EPA 7196 Colorimetric Method
Aluminum (Al)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 202.1, Flame EPA 202.2 or EPA 7020, Furnace
Antimony (Sb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 204.1 or EPA 7040, Flame EPA 204.2 or EPA 7041, Furnace
Arsenic (As)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 206.3 or EPA 7061, Hydride AA EPA 206.2 or EPA 7060, Furnace AA
Barium (Ba)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 208.1 or EPA 7090, Flame AA EPA 208.2, Furnace AA
Beryllium (Be)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 210.1 or EPA 7090, Flame AA EPA 210.2A or EPA 7091, Furnace AA
Boron (B)	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic only	Cool, 4° C	6 months	Curcumin Colorimetric EPA 212.3

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Cadmium (Cd)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 213.1 or EPA 7130, Flame AA EPA 213.2 for EPA 7131, Furnace AA
Calcium (Ca)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 215.1 or EPA 7140, Flame AA
Chromium (Cr)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 218.1 or EPA 7190, Flame AA EPA 218.2 or EPA 7191, Furnace AA EPA 218.3 or EPA 7198, Chelation Extraction
Cobalt (Co)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 219.1 or EPA 7200, Flame AA EPA 219.2 or EPA 7201, Furnace AA
Copper (Cu)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 220.1 of EPA 7210, Flame AA EPA 220.2, Furnace AA
Gold (Au)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 231.1, Flame AA EPA 231.2, Furnace AA
Iron (Fe)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 236.1 or EPA 7380, Flame AA EPA 236.2, Furnace AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Lead (Pb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 239.1 or EPA 7420, Flame AA EPA 239.2 or EPA 7421, Furnace AA
Magnesium (Mg)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 242.1 or EPA 7460, Flame AA
Manganese (Mn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 243.1 or EPA 7480, Flame AA EPA 243.2 or EPA 7481, Furnace AA
Molybdenum (Mo)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 246.1 or EPA 7520, Flame AA EPA 246.2, Furnace AA
Nickel (Ni)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 249.1 or EPA 7610, Flame AA EPA 249.2, Furnace AA
Potassium (K)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 258.1, Flame AA
Selenium (Se)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 270.2 or EPA 7740, Furnace AA EPA 270.3 or EPA 7741, Hydride AA

** **Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Silver (Ag)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 272.1 or EPA 7760, Flame AA EPA 272.2, Furnace AA
Sodium (Na)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 273.1 or EPA 7770, Flame AA
Thallium (Tl)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 279.1 or EPA 7840, Flame AA EPA 279.2 of EPA 7841, Furnace AA
Tin (Sn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 282.1 or EPA 7870, Flame AA EPA 282.2, Furnace AA
Titanium (Ti)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 283.1, Flame AA EPA 283.2, Furnace AA
Vanadium (V)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 286.1 or EPA 7910, Flame AA EPA 286.2 or EPA 7911, Furnace AA
Zinc (Zn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 289.1 or EPA 7950, Flame AA EPA 289.2, Furnace AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

E. GENERAL: MINERAL ANALYSIS/VOLATILES - DRINKING WATER or TITLE 22 CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Complete General Mineral Analysis	1 - 2 liters	Plastic or Glass	Cool, 4° C, additional preservation depends on the analyte list	-----	-----
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1 pH Meter
Alkalinity	50 - 100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA Method 310.1 Titrimetric Method
Calcium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 215.1
Chloride	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA Method 325.3 Titrimetric Method
Copper**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 220.1
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 425.1 Colorimetric
Iron**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 236.1
Magnesium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 242.1
Manganese**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 243.1
Sodium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 273.1
Sulfate	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4 Turbidimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	24 hrs. or filter	EPA Method 120.1 EC Water
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA Method 160.1 Gravimetric
Total Hardness	50 - 100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA Method 130.2 Titrimetric Standard Method 314-A Calculation
Zinc**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 289.1
Volatile Organics (VOAs)	2 - 40 ml containers	Glass, Cap teflon-lined, 40 ml.	No headspace, HCl to pH<2, if residual chlorine then preserve with Na_2SO_4 or Ascorbic Acid Wash. State - No headspace and HCl to pH <2 only	14 days	EPA Method 524.2

** **Each metal can also be analyzed by EPA 200.7 or EPA 6010A.**

F. INORGRANIC ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Arsenic**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 206.3, Furnace AA
Barium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 208.1, Flame AA
Cadmium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 213.1 , Flame AA or EPA 218.2, Furnace AA
Chromium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 218.1, Flame AA EPA 218.2, Furnace AA
Lead**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 239.2, Furnace AA
Mercury	100 ml	Plastic or Glass	HNO_3 to pH <2	28 days	EPA 245.1 ,Cold Vapor
Selenium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 270.2 ,Furnace AA
Silver**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 272.1 ,Flame AA
Nitrate-Nitrogen	50 ml	Plastic or Glass	Cool, 4° C add H_2SO_4 to pH <2 Cool, 4° C	14 days 48 hours	EPA 352.1, Brucine Sulfate EPA 353.3, Cadmium Reduction
Fluoride	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Selective Electrode

**** Each metal can also be analyzed by EPA 200.7 or EPA 200.8.**

G. GENERAL PHYSICAL ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 110.2
Odor	200 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Threshold Odor
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Nephelometric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Acidity	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 305.1, Titrimetric
Alkalinity	50-100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 310.1 ,Titrimetric
Ammonia Nitrogen	100 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 350.1, Colorimetric
BOD	l liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 405.1
Boron	100 ml	Plastic	None	28 days	EPA 212.3 ,Curcumin
Chloride	100 ml	Plastic or Glass	None	28 days	EPA Method 325.3, Mercuric Nitrate or EPA 300.0, Ion Chromatography
COD	20 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA Methods 410.4, Colorimetric
Coliform Fecal Coliform	100 ml	Sterilized Plastic Bottles	Cool, 4° C $Na_2S_2O_3$ preserved for presence of free chlorine	6 hours or 30 hours depending on the test requested	Standard Method 909A or 909C
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 110.2 or 110.3
Cyanide	500 ml	Plastic or Glass	Cool, 4° C NaOH to pH >12	14 days	EPA 335.3, Colorimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 120.1, EC Meter
Fluorides	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Specific Electrode or EPA 300.0, Ion Chromatography
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 425.1, Colorimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Nitrate Nitrogen	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 300.0, Ion Chromatography
Nitrate Nitrite Nitrogen	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	14 days	EPA 353.3, Cadmium Reduction
Nitrite Nitrogen	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 354.1, SPectrophotometric
Odor	200 ml	Plastic or Glass	Cool, 4° C	24 hours	EPA 140.1
Oil and Grease	1 liter	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 413.1, Gravimetric
Orthophosphate	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 365.2, Ascorbic Acid
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1, pH Meter
Phenolics	500 ml	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 420.1, 4-AAP
Radioactivity	1-18 liters	Plastic or Glass Glass only for Tritium	HCl or HNO_3 * to pH <2 *Some exceptions	-----	Standard Method 701
Silica	50 ml	Plastic	Cool, 4° C	28 days	EPA 370.1, Colorimetric Flame AA Method
Sulfates	50 ml - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4, Turbidimetric or EPA 300.0, Ion Chromatography
Sulfides	500 ml	Plastic or Glass	NaOH to pH >9 2 ml Zinc Acetate Cool, 4° C	7 days	EPA 376.1 ,Titrimetric
Sulfites	50 ml	Plastic or Glass	None	Immediate	EPA 377.1 ,Titrimetric
TOC	25 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 415.2, TOC Analyzer
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.1, Gravimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Total Hardness	50-100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 130.2, Titrimetric Standard Method 314-A Calculation
Total Kjeldahl Nitrogen	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4, Ion Specific Electrode
Total Organic Nitrogen TON-TKN-NH_3-N	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4 and EPA 350.3
Total Petroleum Hydrocarbons Scan, by GC	400 ml to 1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days, but this may vary between states so regulations must be consulted	WTPH-HCID by GC/FID or Modified Method 8015, GC
Total Petroleum Hydrocarbons as Gas, by GC	40 ml Approximately 20 grams for a solid sample	Glass	Cool, 4^0 C, Methanol preservation may be a requirement of some states so state regulations must be consulted Cool, 4^0 C	14 days	WTPH-G by purge and trap GC/FID or Modified Method 8015, GC/FID
Total Petroleum Hydrocarbons as Diesel, by GC	1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days	WTPH-D by GC/FID or Modified Method 8015, GC/FID
Total Phosphate	50 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 365.4, Colorimetric
Total Residue	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.3 , Gravimetric
Total Settleable Solids	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 160.5, Imhoff Cone
Total Suspended Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.2, Gravimetric
Total Volatile Solids	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 160.4, Gravimetric
TOX	500 ml	Amber Glass, Teflon Septum	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 450.1 or EPA 9020, TOX Analyzer
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 180.1

APPENDIX 8

Checklist For Analyses With Short Holding Times

CHECKLIST FOR ANALYSES WITH SHORT HOLDING TIMES

To be completed prior to release of release of samples to laboratory

_______ Correct test codes are entered

_______ Dates and times received and collected are correct

_______ Matrices are correct

_______ Sample I.D.s are correct

I certify that all of the above have been checked and were found accurately entered in the LIMS.

______________________________ ______________
(Signature) (Date)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4103

Title: Reviewing a Sample Delivery Group (SDG) and Updating Projqc in the LIMS

Revision History:

Number	Date
0	4/09/96

Written by: Diana Spence
Diana Spence
Date: 4/9/96

Reviewed by: Harry Romberg
Harry Romberg-QC Officer
Date: 4-9-96

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director
Date: 4/9/96

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1. Method Description

1.1.1 The project manager (PM) or their designee reviews the LIMS computer entry versus the chain-of-custody (COC) for accuracy. This should be done as soon as practical and within twenty-four hours (24) of sample receipt.

1.1.2 This method is restricted to use by the person (usually a project manager) who performed the work order transmittal.

1.2. Definition of Terms

1.2.1. SDG - Sample Delivery Group

Projqc - Project QC section of the LIMS. This is where all of the work orders for a specific project are listed with additional information about the QC requirements, TATs and deliverables.

2. Equipment List

2.1. Equipment

2.1.1. The person performing this procedure must have access to a computer which is connected to the LIMS.

3. Operation procedures

3.1. Reviewing the SDG Entry in LIMS

3.1.1 Once a work order has been transmitted, go to the SDG section of the LIMS. This is done by entering [SDG, space, SDG name] at the J(1): prompt. The SDG name is found in the fractions sections of the work order in the SDG field labeled 'SDG#'. **Example** - J(1): SDG HCST4. See Appendix I for an example.

3.1.2 In the SDG verify that all the samples have been entered and that the correct 'fraction' of the SDG (I,G, V, P, etc.). The letters refer to the type of analysis, i.e., Inorganics, GC, Volatiles, and PAHs. These are a few of the types of fractions which might be contained in a SDG. It is not important to know the abbreviations. However, it should be verified that all analyses for each sample can be found in the SDG by looking at the various fractions for that SDG. A fraction may contain more than one analysis. For instance an 'I' fraction (inorganics) might have TOC, Metals and TSS.

3.1.3 When the SDG name is entered at the J(1): prompt, the 'first' page of the first fraction of a SDG will be displayed. The first fraction is determined alphabetically based upon the letter associated with an analysis. For instance, if a work order has analyses for GC analysis (G), metals (I) and PAHs (P), the fractions of a SDG would be listed in LIMS with GC analyses first, metals second, and PAHs last. The information contained on the first page is:

- Laucks sample ID
- QC Designations
- Client ID
- Matrix

3.1.4. Verify that all the samples for that fraction (analysis) have been entered. this is facilitated by entering [F6] which displays the second , third and fourth pages of a fraction. [F6] is a toggle key which pages through the four screens of each SDG entry. The far left hand column of the computer screen will always display the lists of the SDG names with the fractions appendix letter. **Example** - GSI01<u>G</u> (GC fraction), GSI01<u>I</u> (inorganics fractions). See pages 1,2 and 3 of Appendix 1 for examples of each page of a SDG.

3.1.5 The second page of a SDG contains the Laucks samples ID, the VTSR (Verified Time of Sample Receipt) and date collected information.

3.1.6 The third page of a SDG contains the Laucks sample ID and the specific analysis requested. It is important to review this page carefully. If there are many samples in that fraction, they may not all be visible at once. Move down the screen to view all samples by repeatedly pushing the down arrow key, [↓].

3.1.7 Once this fraction has been checked, the other fractions can be checked by using the [F3] (moves the cursor up) and [F4] (moves the cursor down) function keys to place the cursor on the next fraction of the SDG. It is displayed in the column on the left of the screen.. It is possible to view the same page of different fractions by just moving the cursor to the next fraction. For example, if you are in page three of a fraction, when you move to the next fraction, page three of this fraction will be displayed. **[F6] controls the page selection of the SDG, [F3] and [F4] respectively in the far left column.**

3.1.8 If corrections are necessary, return the COC and log-in documents to the Sample Control department. If the person performing the review makes the corrections, the changes must be saved by entering [F8].

3.1.9 To exit the SDG, enter [F1] until the J(1): prompt is displayed.

3.2 Entering a Work Order into 'Projqc' of LIMS

3.2.1 At the J(1): prompt, enter [projqc, space, projqc name]. Example - J(1): projqc OHM_Hawaii. If you are not sure of the projqc name, a name may be entered which is similar. This will bring up the projqc in the vicinity of the name which is desired. The cursor is moved up, [F3], or down, [F4}, to the desired name. The projqc names will be displayed in the far left column of the screen. See page 1 of Appendix II.

3.2.2 Once the correct project name is displayed, move the cursor down to the next empty line in the work order column. enter the work order number. The SDG will appear in the SDG list column. Move the cursor to the matrix columns (W = Water, S = Soil, O = Oil). Under the appropriate column, enter the number of samples for each matrix. If the SDG is to remain open, no other information should be added. Enter [F8] to save the updated SDG.

3.2.3 It the SDG is to be closed, the due date for the data to be submitted to the reporting department and the due date for the hardcopy report to the client must be entered into the appropriate columns. These dates are displayed in Projqc as 'Office' and 'Client' respectively. See page 1 of Appendix II. Enter [F8] to save these changes.

3.2.4 SDG closure is determined by several factors:

- The number of samples in the SDG, per the EPA definition, should not contain more than twenty samples of the same matrix received over a period of not more than 14 calendar days.
- If it is known that more samples will be arriving for the same project in less than fourteen days, and the SDG is not full, it may be desirable to maintain the SDG in 'open' status until arrival of the next samples.

- If the client desires a fast TAT for the hardcopy package, the SDG should be closed regardless of the number of samples in it.

These are judgment decisions with the exception of the EPA definition of an SDG), which must be made by the project manager.

3.2.5 Once the SDG has been closed, send e-mail notification of the closure to all departments affected. This would be any department involved with the analyses for the work order, Sample Control and the reporting department.

3.2.6 Finally, the paperwork is submitted to the reporting department detailing the nature and status (open or closed) of an SDG. The forms submitted would be the following, arranged in the order listed below:

- Pre-package checklist
- Chain-of-Custody forms (original or top, white copy)
- * Sample Receipt Log (1) CLP (original)
- * Supplemental Sample Receipt Log (original)

* A copy of each of these forms must be made and attached to the copy of the COC for the transmittal.

See Appendix III for examples of these forms.

3.3 Creating a New Projqc

3.3.1 When a new project commences, it may be necessary to create a new entry in Projqc. The factors which would determine if this is necessary may include the following:

- The complexity of the project
- The duration of the project
- How many laboratory departments are affected by the project

Projqc entries are applicable to both CLP and non-CLP project. The information contained in this section of the LIMS is accessible by all laboratory staff who have rights to the LIMS and is a valuable form of communication for project requirements. However, it does not take the place of kick-off meetings for projects or e-mail regarding specific samples problems.

3.3.2 To create a new projqc entry, enter [projqc] at the J(1): prompt. This will display the first projqc entry in the LIMS. The cursor will be in the field called 'Proj Name:'. Enter the name of the new projqc entry. this name may consist of up to ten characters, alpha and numeric, and one underscore in the place of a hyphen, if desired. Once entered, push [Enter]. The cursor will move to the next field, 'Client Code'. See page 1 of Appendix II.

3.3.3 In the 'Client Code' field, enter the correct client code for this work order. This can be found in the work order. Push [Enter]. The cursor will next appear in the 'Desc:' field.

3.3.4 In this field enter a brief description (one cryptic sentence) of the project. Push [Enter]. the cursor will next appear in the bottom line of the projqc header. Enter the correct information for these questions (QAPP?, pH/Temp?). This would usually be 'yes' if the project is CLP or USACE (US Army Corps of Engineers).

3.3.5 Due to software complications, in the field 'Sort by (W/S):' it is best to enter [N].

3.3.6 Enter [F8] to save this new projqc. Item 3.3.1 through 3.3.5 would be considered the header information for a projqc. This is page 1 of the projqc. It is here that the work orders for that project will be entered when the samples arrive at the laboratory.

3.3.7 Next go to page 2 of the projqc. This is accomplished by pushing [F6]. The header information will already be there. The information which must be entered here is:

- Results TAT (Fax)
- Report TAT (hardcopy)
- Deliverables - CLP, LTL (Laucks Testing Laboratory or non-CLP)
- MPR - Monthly Progress Report (required for HAZWRAP projects)
- EDD deliverables - CLP or LTL (any non-CLP, custom EDD)
- Deliverables Comments - Any special project comments
- Penalties - enter any applicable penalties here

Enter [F8] to save this information.

3.3.8 Next go to page three of the projqc. This is accomplished by entering [Ctrl,F6]. of a project can be written as an overview. See page three of Appendix II. The information contained here is:

- Client name
- Project Name
- Overview
- Schedule
- Analytes
- Protocol
- QC
- Turnaround
- Penalties
- CRDLs
- Holding Times
- Deliverables
- Additional Comments

After entering this information, enter [F8] to save it. It may be advantageous to enter [F8] periodically as the information is entered to prevent loss in the event of a power surge.

Appendix I

Example of SDG Entry in LIMS

GSI01
KEYS
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P
GSI02V
GSI03G

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/
Fraction : G SPVMITO Updated: 10/23/
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
PROJQC ID : GS_CHANUTE Max. Samps: 21

Work Ord	Samp Num	QC	Client ID	Mat	TS	pH	Associated Blank
9510423	01		B952/SB-952-1/SS4,6-8/LK	S	88		B1018GSVSL
9510423	02		B952/SB-952-1/SS9,16-18/LK	S	89		B1018GSVSL
9510423	03		B952/SB-952-2/SS4,6-8/LK	S	83		B1018GSVSL
9510423	04		B952/SB-952-2/SS5,8-10/LK	S	84		B1018GSVSL
9510434	01		B952/SB-952-10/SS8,14-16LK	S	84		B1018GSVSL
9510434	02		B952/SB-952-3/SS3,4-6/LK	S	84		B1018GSVSL
9510434	03	MS	B 52/SB-952-3/SS6,10-12/LK	S	89		B1018GSVSL
9510434	05	BMS	CH-5092500-S-SPOON-ERB-1	W			B1017GSVWL
9510434	06		CH-POT-FB-1	W			B1017GSVWL
9510434	07		CH-ASTM-5092500-FB-1	W			B1017GSVWL
9510459	01		B952/SB-952-4/SS4,6-8/LK	S	84		B1018GSVSL
9510459	02		B952/SB-952-4/SS7,12-14/LK	S	89		B1018GSVSL
9510459	03		B952/SB-952-5/SS4,6-8/LK	S	87		B1018GSVSL
9510459	04		B952/SB-952-5/SS6,10-12/LK	S	87		B1018GSVSL
9510459	08		B952/SB-952-6/SS4,6-8/LK	S	84		B1018GSVSL
9510459	09		B952/SB-952-6/SS6,10-12/LK	S	90		B1018GSVSL
9510497	01		B952/SB-952-7/SS3,4-6/LK	S	89		B1018GSVSL
9510497	02		B952/SB-952-7/SS8/4-16/LK	S	91		B1018GSVSL
9510540	01		B952/SB-952-8/SS3,4.0-6.0	S	90		B1018GSVSL
9510540	02		B952/SB-952-8/SS6,10.0-12	S	90		B1018G[illegible]

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: ______ Case Number: _____

Work Ord Samp Num	VTSR	Date Collected
9510423-01	10/11/95	10/10/95
9510423-02	10/11/95	10/10/95
9510423-03	10/11/95	10/10/95
9510423-04	10/11/95	10/10/95
9510434-01	10/12/95	10/11/95
9510434-02	10/12/95	10/11/95
9510434-03	10/12/95	10/11/95
9510434-05	10/12/95	10/10/95
9510434-06	10/12/95	10/10/95
9510434-07	10/12/95	10/10/95
9510459-01	10/13/95	10/11/95
9510459-02	10/13/95	10/11/95
9510459-03	10/13/95	10/11/95
9510459-04	10/13/95	10/11/95
9510459-07	10/13/95	10/12/95
9510459-08	10/13/95	10/12/95
9510459-09	10/13/95	10/12/95
9510497-01	10/14/95	10/12/95
9510497-02	10/14/95	10/12/95
9510540-01	10/17/95	10/15/95
9510540-02	10/17/95	10/15/95

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: ______ Case Number: _____

Work Ord	Samp Num	Fractions JP4										
9510423-01		X										
9510423-02		X										
9510423-03		X										
9510423-04		X										
9510434-01		X										
9510434-02		X										
9510434-03		X										
9510434-05		X										
9510434-06		X										
9510434-07		X										
9510459-01		X										
9510459-02		X										
9510459-03		X										
9510459-04		X										
9510459-07		X										
9510459-08		X										
9510459-09		X										
9510497-01		X										
9510497-02		X										
9510540-01		X										
9510540-02		X										

Comments:

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19
Desc: UST sites Updated: 01/22
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Ord#	Last Date	SDG#	Office	Client	Matrix W	Matrix S	Matrix O
9512516	12/19/95	HI033	01/31/96	02/02/96	2	3	
9601349	01/22/96	HI034	02/20/96	02/22/96	1	7	
9601351	01/10/96	HI035			3		
9601587	01/22/96	HI034	02/20/96	02/22/96	2	6	

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/
Desc: UST sites Updated: 01/22/
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Results turnaround 7 WORK.DAYS
Report turnaround 30 CAL.DAYS

Deliverables Penalties
LTL report
"CLP" report X
MPR required
CLP disk
LTL disk
Other
Other

Deliverables comments

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Client : OHM Remediation Services, Inc.

Project Name: UST Sites in Hawaii

Overview : 90% project consists of Soil samples, 10% water/rinsate samples

Schedule : Delivery of samples over 6 months. Weekly schedule will be obtained one week in advance of sample receipts.

Analytes : Water
29- BTE samples via 8020
36- 8015 Mod (TPH gas or diesel or 418.1)
17- 6010 for Pb, Cd, Cr
30- 8310 Naphthalene, Acenapnthene, Fluoranthene, and low level Benzo(a)pyrene
7- 8080 Low level PCBs 2L to 1mL
7- 8010 tetrachloroethylene, and 111-trichloroethane
10- 1020 flammability
1- HPLC (method supplied by client 2/16) for PGDN and 2-nitrodiphenylamine, Samples to arrive after March
5- 6010 Pb only

Soil
260- BTE samples via 8020
327- 8015 Mod (TPH gas or diesel or 418.1)
152- 6010 for Pb, Cd, Cr
264- 8310 Naphthalene, Acenaphthene, Fluoranthene, and Benzo(a)pyrene
60- 8080 PCBs
7- 8010 tetrachloroethylene, tand 111-trichloroethane
10- 1020 flammability
3- HPLC (method supplied by client 2/16) for PGDN and 2-nitrodiphenylamine, Samples to arrive after March
40- 6010 Pb only

Protocol : SW-846 with NEESA Level C data package. See me if you need a copy of the NEESA Requirements. I have the June 38 version. Site specific QC required. Batch in SDGs.

QC : Yes! See handout submitted to; JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,BO

Turnaround : FAX 7 working days from sample receipt Hard Copy 30 calendar days from SDG closure EDD Not applicable

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Penalties : See PROJQC, page 2

CRDLs : Yes! See minimum detection limits in handout submitted to; JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,SO

Holding Time: Routine SW-846 from collection.

Deliverables: NEESA Level C

Comments :

All stock pile samples must be thoroughly homogenized prior to analysis.

UPDATES will be added (with the date) to this file.

Pam, see MarkS regarding special test codes for low level metals prep! Something like, LXWM1 for the digestion. Also, these samples will be batched in SDGs. See MarkS.

First tentative schedule starting 3/2 or 3/3:

90 soils - BTE (8020) - X
40 soils - TPH Gas
50 soils - TPH Diesel OR 418.1
50 soils - 8310 PAHs
40 soils - Pb only (6010)

3/15/95 sh;

Art Taddeo requested pricing for TCLP Pb, Cd, and Cr (to comply with TCLP regulations). I provided pricing for soils at $130.00 ea.

10/26/95 ds

Data packs are to be sent to:

Kim Osgood
OHM
20015 72nd Ave.S
Kent, WA 98032

Reports are to be faxed to Bob Rooks at 808-682-1880 in HI.

G

KEYS
GLS08O
GLS08P
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/9
Fraction : I SPVMITO Updated: 10/17/9
Project : AFCEE/Chanute 952 Client: GSI (Inorganic)
PROJQC ID : GS_CHANUTE Max. Samps: 21

Comments:
Use Control F6 for more PROJQC details.

F7=Del,/n,Name 8=Write,/n,In_use 9=Print,Db_enter?,Keys? 10=More,Help

Appendix II

Example of Projqc in LIMS

Appendix III

Example of SDG Paperwork

PRE-PACKAGE CHECKLIST

SDG# GTI27 open ☐ closed ☑

Work Order #(s)	Fax Due Date(s)
960212	1/16/96

Due to Office: 2/7/96

Due to client: 2/9/96

E.D.D.: yes ☐ Custom or CLP *(circle one)* no ☑

If yes, can the package ship without the disk? ______

Full Deliverables? yes ☑ no ☐

If no, what forms/raw data are required? ______________________

No. of Copies: 1

Special package requests:

Assigned Document controller: ____________

THIS INFORMATION WILL BE USED FOR REPORTING/BILLING (SEE BELOW)

CHAIN OF CUSTODY RECORD 96Ø1313

Laucks Testing Laboratories, Inc.

NAME: Groundwater Technology GSI

ADDRESS: 19033 W Valley Hwy D-101, Kent WA 98302

ATTENTION: Diana Spence

PROJECT NAME: Bangor Site A

PROJECT CONTACT: Stan Thompson

TELEPHONE/FAX: 360 779 9564 / 360-779-1676

JOB/P.O. NO.: 83001-2153 110

SAMPLER (SIGNATURE) (PRINTED NAME) Daryl Brown

WORK ORDER ID: 85-01-2153 PAGE 1 OF 1

DATE: 1-8-96

SUBMITTED AT:

TESTS TO PERFORM: M8330

LAB SAMPLE	SAMPLE ID / LOCATION	DATE	TIME	M8330	NO. OF CONTAINERS	OBSERVATIONS, COMMENTS, SPECIAL INSTRUCTIONS
1)	INF-CL-209-010896	1-8-96	0700	2L	2L	Fax Results to 360-779-1676

TOTAL NO. OF CONTAINERS: 2L

INSTRUCTIONS
1. USE ONE LINE PER SAMPLE.
2. BE SPECIFIC IN TEST REQUESTS.
3. CHECK OFF TESTS TO BE PERFORMED FOR EACH SAMPLE.

BILLING INFORMATION, IF DIFFERENT THAN ABOVE
NAME / ADDRESS / ATTN: / CITY, STATE, ZIP

TURNAROUND REQUEST
- [ ] 24-48 HRS (100% SUR)
- [x] 5 DAYS (50% SUR)
- [ ] STD 10-14 DAYS
- [ ] OTHER

CHAIN OF CUSTODY SEALS? [ ] YES [ ] NO [ ] NA

SHIPPED VIA [ ] UPS [ ] FED EX [ ] BUS [ ] HAND [ ]

TEMPERATURE

RELINQUISHED BY (SIGN AND PRINT)	DATE / TIME	RECEIVED BY (SIGN AND PRINT)	DATE / TIME
Daryl Brown	1-8-96 1600	Pony Express / PAM R. JOHNSON	1/9/96

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (1) CLP

P:\PM\Holly\docs\...\memo.doc

Initial once samples are checked in ______

DATE RECEIVED: 1/9/96
TIME RECEIVED: 0726
CLIENT NAME: GSI
SDG #: GTE27
COC #: N/A

SAMPLE LOG-IN DATE: 1/9/96
WORKORDER #: 9601313
CLIENT PROJECT: Bangor Site A
AIRBILL ATTACHED?:(#) 2576393
RECEIVED BY: B

Non-Conformance: (Check applicable item(s)) Client IDs affected:

- ☐ (1) Not enough sample sent for proper analysis. #s affected: ______
- ☐ (2) Sample Bottle received broken and/or cap not intact. ______
- ☑ (3) Custody seal: Absent ____ Present/Intact ✓ Present/Broken ____
- ☐ (4) Any temperature out of compliance: ______
- ☐ (5) Sample received outside of holding time. ______
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used. ______
- ☐ (7) Illegible sample numbers or label missing from bottles. ______
- ☐ (8) Identification on bottle same as identification on paperwork: yes: ____ no: ____
- ☐ (9) Incomplete instructions received with sample(s), i.e.,
- ☐ no Request for Analysis, no Chain-of-Custody. ______
- ☐ (10) Samples received in improper container. ______
- ☐ (11) Samples held in field before receipt by Lab. Days (specify) ______
- ☐ (12) Air Bubble(s) in ___ of ___ samples for volatiles analysis. ______
- ☐ (13) Other ______

CORRECTIVE ACTION: (Check applicable item(s))

Correction action taken by:

		Initials	Date
☐	(1) Client informed verbally (Client Services).		
☐	(2) Client informed by memo/letter/fax (Client Services).		
☐	(3) Sample processed "as received" (Sample Entry).		
☐	(4) Re-sampling requested of client (Client Services).		
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).		
☐	(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).		
☐	(7) Other (Specify) ______		

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

Work Order Number: 9601313
Assigned SDG Number: [illegible]

Temperature		pH of Bottle Types								
		8330								
1	20123	2)7								

llowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

emperature	Allowable temperature range is 4± 2 degrees Celsius
cid Preserved pH	pH must be less than 2
ase Preserved pH	pH must be greater than 12

TMPLOG.DOT 2.0

LAUCKS TESTING LABORATORIES INC.
Seattle. Washington

SOP #:LTL-4201

Title: **Package Deliverables for all Reporting Levels**

Revision history:

Number	Date
000	1/31/96

Written by: ______________________ Date: 1-31-96
Tom Marino

Reviewed by: Charlene Nix ______________________ Date: 1-31-96
Charlene Nix

Reviewed by: Harry Romberg ______________________ Date: 1-31-96
Harry Romberg, QC Officer

Approved by: Karen J Kotz ______________________ Date: 1/31/96
Karen Kotz, Laboratory Director

UNCON[illegible]D

Table of Contents

1. Introduction and Scope

1.1 This SOP descibes the contents of the different levels of reports at Laucks Testing Laboratories, Inc.

1.2 Complete CLP-type data package (level IV):

A complete data package (level IV) submitted to a client consists of a cover page, a narrative, chain-of-custody copies, an index, and a separate section for each analytical fraction containing all forms and raw data. The entire package is paginated sequentially beginning with #1.

1.3 Forms only data package (level III):

A "forms only" data package (level III) submitted to a client contains all of the above with the exception of the raw data.

1.4 Database Report (level II):

A database report (level II) contains forms generated from the database and includes many of the forms in a level III or IV package. The contents are indicated in Appendix I and are not described further.

1.5 Paper Job Report (level I):

A "paper job" report is created for special chemistry and food chemistry and usually contains a format specified by the client or results only. The contents are indicated in Appendix I and are not described further.

2. Description of Contents of Level III and IV packages

Detailed below are the elements that may be included in a level III or level IV package. Appendix I contains tables that signify whether an element is incorporated into a final report. Client specific requests may dictate that some elements may be added or deleted and these are documented during project initiation in the LIMs system.

2.1 Cover Page

The package cover page will contain the laboratory name, client name, work order numbers, SDG number and the date.

2.2 Narrative

2.2.1 Sample receipt and Identification:

This section lists all client sample names, the corresponding laboratory sample names and the analyses requested for each sample. (The analyses are generally abbreviated to three letters.)

2.2.2 Analytical request key:

This section defines the abbreviations listed in the above section.

2.2.3 Sample Identification on Forms:

This sections is used to explain any abbreviations to client sample names on any of the forms (occasionally forms software does not accommodate lengthy client sample I.D.s).

2.2.4 General remarks on organic analysis:

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested organic fractions.

2.2.5 Specific remarks on organic analysis:

These are comments written for each organic analytical fraction describing any anomalies, deviations from the specified method, dillutions, holding time violations, corrective actions etc. These comments are written by the respective analysts.

2.2.6 General remarks on inorganic analysis

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested inorganics fractions.

2.2.7 Specific Remarks on inorganic analysis:

These are comments written for each inorganic analytical fraction describing any anomalies,deviations from the specified method, dilution's, holding time violations,corrective actions etc. These comments are written by the respective analysts.

2.2.8 Release of data:

This page is signed by both the respective project manager and the technical director. also contained on this page is information on who to contact regarding specific questions as well as the laboratory telephone and fax numbers

2.3 Chain-of-Custody Copies:

This section contains the chains of custody received with the samples as well the laboratory receipt and temperature logs.

2.4 Index:
The index should list all data fractions and sub-fractions with the corresponding page numbers.

2.5 Organic fractions

Level IV Organic data packages are subdivided into five sections: QC Summary, Sample Data, Standards Data, Raw QC Data, and Bench Sheets.

Level III (forms only) organic data packages contain only the **forms** from these sections. No bench sheets or raw data are provided.

Only Volatiles, Semi-volatiles and Pesticides/PCBs have official CLP form numbers and protocol. Every effort is made to ensure that the same information appears on forms for all other fractions.

NOTE: the form numbers that appear below are seen only on forms for Volatiles, Semi-Volatiles and Pesticides/PCBs. Forms for all other fractions contain the same information but no actual form numbers.

2.5.1 QC Summary:

The QC Summary contains the following forms:

a.) Form II: Surrogate recovery report
b.) Form III: MS/MSD Recovery report
c.) Blank spike report
d.) Form IV: Method blank summary
e.) Form V: Tuning and Mass Calibration Standard
f.) Form VIII: Internal standards Area Summary

2.5.2 Sample Data:

Sample data contains the following forms and data

a.) Form I (analysis data sheet) including TICs
b.) Raw Data

2.5.3 Standards Data:

The standards data below are divided into two formats: Volatile/Semi-Volatile and Pesticide/PCB. Every effort is made to ensure that forms for all other fractions adhere closely to whichever of the two formats is most applicable.

2.5.3.1 Volatile/Semi-Volatile

a.) Form VI and Initial Calibration Data
b.) Form VII and Continuing Calibration Data

2.5.3.2 Pesticide/PCB

a.) Form VIII: Pesticide Analytical Sequence
b.) Form IX: Pesticide/PCB Standards Summary
c.) Form X: Pesticide/PCB Identification (positive results)
d.) Pesticide standard chromatograms and data system printouts for Evaluation of standard mix A,B, and C
e.) Pesticide.standard chromatograms and data system printouts for individual standard mix A, and B
f.) Pesticide Standard Chromatograms and data system printouts for all multi-response pesticides/PCBs and quantitation standards
g.) A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

2.5.4 Raw QC Data:

The Raw QC Data below are divided into three formats: Volatile, Semi-Volatile, and all other fractions.

2.5.4.1 Volatile
- a.) BFB
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.2 Semi-Volatile
- a.) DFTPP
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.3 All other fractions
- a.) Blank Data
  - 1.) Form I
  - 2.) Raw data

b.) Matrix Spike Data
1.) Form I
2.) Raw data
c.) Matrix Spike Duplicate Data
1.) Form I
2.) Raw data

2.5.5 Bench Sheets:

The bench sheets section contains any miscellaneous paper work such as log book pages, in-house tracking sheets, in-house chains of custody, extraction bench sheets etc.

2.6 Inorganics Fractions:

There are two kinds of inorganics fractions: Metals and Miscellaneous Inorganics (conventionals).

2.6.1 Metals

2.6.1.1 Cover Page:

This page lists all samples analyzed and is signed and dated by the analyst

2.6.1.2 Inorganics Analysis Data Sheet
a.) forms I

2.6.1.3 Quality Control Data
a.) Form II (part 1): Initial and Continuing Calibration Verification
b.) Form II (part 2): CRDL Standard for AA and ICP
c.) Form III: blanks
d.) Form IV: ICP Interference Check Sample
e.) Form V (part 1): Spike Sample Recovery
f.) Form V (part 2): Post Digest Spike Sample Recovery
g.) Form VI: Duplicates
h.) Form VII: Laboratory Control Sample
i.) Form VIII: Standard Addition Results
j.) Form IX: ICP Serial Dilutions
k.) Form XIII: Preparation Log
l.) Form XIV: Analysis run Log

2.6.1.4 Quarterly Verification of Instrument Parameters

a.) Form X: Instrument Detection Limits (quarterly)
b.) Form XI (part 1): ICP Inter element Correction Factors (annually)
c.) Form XI (part 2): ICP Inter element Correction Factors (annually)
d.) Form XII: ICP Linear Ranges (quarterly)

2.6.1.5 Raw Data

a.) ICP
b.) Graphite Furnace
c.) Mercury
d.) Cyanide

2.6.1.6 Digestion and Distillation Logs

a.) ICP
b.) Graphite Furnace
c.) Mercury
d.) Cyanide

2.6.2 Miscellaneous Inorganics

2.6.2.1 Cover Page

This page lists all samples analyzed and is signed and dated by the analyst.

2.6.2.2 Inorganics Analysis Data Sheet

2.6.2.3 Quality Control Data

a.) Preparation blanks database Report
b.) MS/MSD Report
c.) MS/Duplicate Database
d.) SRM Report

2.6.2.4 Raw Data

The Miscellaneous Inorganics raw data is divided into sections by analytes.

Appendix I

Laucks Testing Laboratories
Organics
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Surrogate Rec.		Y	Y	Y
Method Blank		Y	Y	Y
Blank Spike		Y	Y	Y
MS/MSD		Y	Y	Y
Tune			Y	Y
Initial Calib.			Y	Y
Cont. Calib.			Y	Y
IS Area				Y
Raw Data			Optional	Y
Chromatograms			Optional	Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

Laucks Testing Laboratories
Metals
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	Y
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Method Blank		Y	Y	Y
SRM/LCS/BS		Y	Y	Y
MS/Dupe		Y		Y
MS/MSD			Y	Y
ICV			Y	Y
ICB			Y	Y
CCV			Y	Y
CCB			Y	Y
CRA			Y	Y
CRI			Y	Y
Post Spike			Y	Y
GFAA MSA			Y	Y
ICP Ser. Dil'n.			Y	Y
IDLs			Y	Y
Interelement Corr. Factors				Y
Linear Range				Y
Prep Log				Y
Run Log				Y
Raw Data				Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

Laucks Testing Laboratories
Wet Chemistry
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	Y
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Method Blank		Y	Y	Y
SRM/LCS/BS		Y	Y	Y
MS/Dupe		Y		Y
MS/MSD			Y	
ICV			Y	Y
ICB			Y	Y
CCV			Y	Y
CCB			Y	Y
Raw Data				Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7003

Title: **Inorganic Glass Cleaning Procedure**

Revision history:

Number	Date
2	04/09/97
1	01/05/88

Written by: Bill Lundberg Date: 4/9/97
Bill Lundberg, Inorganics Supervisor

Reviewed by: Harry Romberg Date: 4-9-97
Harry Romberg, QA Officer

Approved by: Karen J. Kotz Date: 4/9/97
Karen Kotz, Laboratory Director

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Table of Contents

1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to define the procedures used in the inorganics department for the cleaning of glassware. The objective is to define a specific method of cleaning that is adapted to both the substances that are to be removed, and the determination to be performed.

1.2 Definitions

1.2.1 DIW = deionized water

2. Equipment List and Standards

2.1 Standard laboratory glassware, including but not limited to:

2.1.1 Volumetric flasks
Beakers
Funnels
Separatory funnels
Kjeldahl flasks
Nessler tubes
Erlenmeyer flasks
Burets
BOD bottles
Distillation Apparatus

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Several cleaning procedures or soaking procedures require the use of a chromic acid cleaning solution, concentrated HNO_3 or H_2SO_4. USE APPROPRIATE SAFETY PRECAUTIONS FOR ACID USE! Wear safety glasses, lab coat, and gloves.

3.1.2 Some oily samples or profile samples may require the use of acetone or chloroform to clean the glassware. AGAIN, USE APPROPRIATE SAFETY PRECAUTIONS FOR SOLVENT USE! Wear safety glasses, lab coat, and gloves. Dispose of solvent waste in appropriate solvent waste barrel.

3.1.3 Some profile-type samples may not clean up even with solvent washes, and therefore may need to be baked in the muffle furnace at 500 degrees C for 1-3 hours.

3.1.4 CAUTION: Be sure to evaporate any residual solvent from glassware before putting in muffle furnace.

3.1.5 Do not put soft glass (non-pyrex, kimax, etc.) in a muffle furnace or it will shatter. Take appropriate precautions with hot materials.

4. Operation procedures

4.1.1 All glassware must be scrupulously cleaned. The analyst that performs each specific analysis is responsible for the proper cleaning of his or her own glassware. Glassware used in routine analysis is kept separate from the general use glassware. Specific cleaning procedures are listed by type of analysis.

4.1.2 ALKALINITY

4.1.2.1 Glassware Erlenmeyer flasks

4.1.2.2 Buret

4.1.2.3 Cleaning Procedures - Rinse with DIW.

4.1.2.4 Air dry.

4.1.3 AUTO ANALYZER (refer to the applicable analytical SOP)

4.1.4 BOD

4.1.4.1 Glassware BOD bottles

4.1.4.2 Glass pipettes

4.1.4.3 Cleaning procedure - Wash with hot tap water and Alconox.

4.1.4.4 Rinse with hot tap water.

4.1.4.5 Rinse with DIW.

4.1.4.6 Air dry.

4.1.5 COD

4.1.5.1 Procedure for Glassware Condensers, Erlenmeyer flasks, Buret

4.1.5.2 Cleaning Procedure - Rinse well with DIW only.

4.1.5.3 Soaking Procedure - Acid soak flasks with C.O.D. acid for 10 minutes prior to use. Rinse well with DIW.

4.1.6 CYANIDE

4.1.6.1 Procedure for Glassware, Volumetric flasks, Distillation apparatus

4.1.6.2 Cleaning procedure for Flasks - DIW rinse only.

4.1.6.3 Distillation apparatus - occasional H_2SO_4 and DIW wash, but generally DIW rinsed only.

4.1.7 Hexavalent Chromium

4.1.7.1 Cleaning procedure - Wash with hot tap water and Alconox.

4.1.7.2 Rinse well with DIW.

4.1.7.3 Air dry.

4.1.8 FORMALDEHYDES

4.1.8.1 Procedure for GlasswareBeakers, Erlenmeyer flasks, Volumetric flasks, Test tubes

4.1.8.2 Cleaning procedures - ,Rinse well with DIW only.

4.1.8.3 Never contaminate glassware with HNO_3 .

4.1.9 HARDNESS

4.1.9.1 Procedure for Glassware, Erlenmeyer flasks, Buret

4.1.9.2 Cleaning procedure - Rinse with DIW only.

4.1.10 KJELDAHL NITROGEN-LOW LEVELS (TKN AND AMMONIA)

4.1.10.1 Procedure for Glassware, Kjeldahl distillation apparatus, Kjeldahl flasks, Erlenmeyer, flasks or beakers

4.1.10.2 Cleaning procedure for Kjeldahl flasks - pre-distill with DIW and NaOH, and do final DIW rinses.

4.1.10.3 Beakers or Erlenmeyer flasks- DIW rinse only.

4.1.11 OIL AND GREASE

4.1.11.1 Procedure for Glassware, Volumetric flasks. Beakers, Funnels, Separatory funnels, Soxhlet digestion apparatus

4.1.11.2 Cleaning procedure for Vol. flasks, funnels, soxhlet apparatus- freon rinse 3-4 times.

4.1.11.3 Cleaning procedure for Beakers, separatory funnels- hot tap water and Alconox, rinse well with hot tap water, and dry.

4.1.12 METALS (INCLUDING HYDRIDES AND MERCURY)

4.1.12.1 Procedure for Glassware, Volumetric flasks. Beakers, Erlenmeyer flasks, BOD bottles, Digestion caps or watchglasses

4.1.12.2 Cleaning procedure - Rinse well with DIW only.

4.1.12.3 OPTIONAL:

4.1.12.4 If oily or difficult to clean, wash with acetone. scrub with hot tap water and Alconox, and rinse well with DIW.

4.1.12.5 Concentrated acid wash and DIW rinsed.

4.1.12.6 Bake in muffle furnace at 550 degrees C for 1 hour and rinse well with DIW.

4.1.12.7 NOTE: Some metals glassware should not be cleaned with Alconox at all.

4.1.12.8 Soaking procedure - Acid soak all metals glassware in HNO_3 and DIW.

4.1.12.9 Fill glassware with DIW and add 4-8 mls HNO_3. Cover and store until next use.

4.1.13 PHENOL

4.1.13.1 Procedure for Glassware, Kjeldahl flasks, Erlenmeyer flasks

4.1.13.2 Cleaning procedure for Kjeldahl flasks - pre-distill with H_2SO_4 and catalyst, DIW rinse.

4.1.13.3 Cleaning procedure for Erlenmeyer flasks - acid H_2SO_4 wash and DIW rinse.

4.1.13.4 NOTE: Use only those Kjeldahl flasks designated for use in phenol distillations.

4.1.14 PHOSPHATE

4.1.14.1 Cleaning procedure for GlasswareBeakers

4.1.14.2 Acid wash with H_2SO_4 .

4.1.14.3 Rinse with DIW.

4.1.15 TOC (SOILS)

4.1.15.1 Glassware, TOC combustion boats

4.1.15.2 Cleaning procedure - Brush out last sample.

4.1.15.3 Incinerate boat in muffle furnace at 950 degrees C.

4.1.16 TOX

4.1.16.1 Cleaning procedure for Glassware, Volumetric flasks, Misc. TOX glass parts

4.1.16.2 Cleaning procedure - Soak in chromic acid solution.

4.1.16.3 Wash with hot tap water and alconox.

4.1.16.4 Rinse well with DIW.

4.1.16.5 Bake in muffle furnace at 400 degrees C.

4.1.16.6 Cool.

4.1.16.7 Store in glass teflon sealed container inside a dessicator.

5. References

5.1.1 See the applicable analytical or preparation SOP for specific cleaning issues and references.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7015

Title: **Acid Digestion of Sediments, Sludge, and Soils by SW846 Method 3050B**

Revision history:

Number	Date
0.0	1/18/99

UNCONTROLLED

Written by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 1-20-99

Reviewed by:
Ming-Hwa Hwang, Metals Supervisor
Date: 1-20-99

Approved by: Harry Romberg
Harry Romberg, QA Officer
Date: 1-21-99

Table of Contents

1. Introduction and Scope

1.1. This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP) or inductively coupled argon plasma spectroscopy - mass spectrometry (ICP-MS). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also step 2.1):

FLAA/ICP-AES		GFAA/ICP-MS
Aluminum	Magnesium	Arsenic
Antimony	Manganese	Beryllium
*Arsenic	Molybdenum	Cadmium
Barium	Nickel	Chromium
Beryllium	Potassium	Cobalt
Cadmium	*Selenium	Iron
Calcium	Silver	Lead
Chromium	Sodium	Molybdenum
Cobalt	Thallium	Selenium
Copper	*Tin (FLAA only)	*Tin (ICP-MS only)
Iron	Vanadium	Thallium
Lead	Zinc	

* Although Arsenic and Selenium (analyzed by FLAA/ICP) and Tin (analyzed by FLAA or ICP-MS) are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3050B, Laucks has demonstrated adequate recovery for these analytes from this digestate.

1.2 It should be noted that LTL-7015 deviates from SW846 Method 3050B in one instance; however, Laucks has demonstrated adequate recovery for all analytes listed above using this method. The modification implemented in LTL-7015 is outlined as follows:

- In place of adding 30% H_2O_2 in 1-mL aliquots as outlined in Section 7.2.2 of SW846 Method 3050B, seven 1-mL aliquots of 30% H_2O_2 are added in Section 6.2 of LTL-7015.

2. Summary of Method

2.1 A representative 1. to 1.5g sample is digested in nitric acid and hydrogen peroxide. The digestate is then either reduced to a low volume or refluxed with hydrochloric acid. Hydrochloric acid is used for flame AA and ICP analyses only. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, (2) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and Zn. The hydrochloric acid step is omitted for the furnace AA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and Tl. The diluted samples have an approximate acid concentration of 5% (v/v).

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2. These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3. Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before taking any breaks, eating, or going home for the day.

3.4. Fertilizer samples are chemically complex matrices and should be handled with extreme caution. Many components found in fertilizer are highly reactive to acid and all acid additions should be made with special care, in a hood, standing back from the sample. Also, these samples have been known to "pop" or explode during heating, so care should be taken not to stand close to or have any body part over the samples while they are being heated.

4. Equipment

- 150-mL beakers or equivalent, acid-washed
- Analytical balance capable of accurately weighing to the nearest 0.01 g
- Watch glass for beakers, acid-washed
- Eppendorf or other micropipets
- Hot plate or equivalent heating source, adjustable and capable of maintaining a temperature of 90-95° C

- 100-mL graduated cylinder, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Glass funnels, acid-washed
- 100-mL volumetric flasks, acid-washed
- Sample digestate bottles, acid-washed
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, SOP #: LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free deionized water. All references to water in the method refers to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better

5.3 Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better

5.4 Hydrogen peroxide (30%), H_2O_2, ACS Reagent grade or better

The method blank prepared along with the samples is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion logs.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions

6.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh 1.00-1.50 g of sample to the nearest 0.01 g directly into an Erlenmeyer flask. For samples with low percent solids, a larger sample size may be used to attain the equivalent of 1.00-1.50 g dry basis as long as digestion is completed. Add appropriate spiking mix aliquots to QC samples at this point.

6.2 Add 5 mL of water and 5 mL of concentrated HNO_3, and mix the slurry, place the watch glasses on the beakers, Heat the samples to 95° C and reflux without boiling for 15 minutes. Record the temperature achieved during the digest on the digest log. Allow the samples to cool, then add 5 mL of concentrated HNO_3. Heat the sample to reflux for 30 minutes. Repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample indicating the complete reaction with HNO_3. Reduce the volume to 5 mls. Allow the samples to cool, add 2 mL water and 3 mL 30% H_2O_2, and reflux the samples for 10 minutes. Cool the samples, add 3 mL 30% H_2O_2, and reflux the samples until effervescence subsides. Cool, add seven 1 mL aliquots of 30% H_2O_2 separately, and reduce volume to 5 mls. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence after the peroxide additions.

6.3 A. If the sample is being prepared for (a) the ICP analysis of As and/or Se, (b) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and/or Zn, then add 10 mL of concentrated HCl, return the flask to the hot plate, and reflux for an additional 15 minutes without boiling. After cooling, dilute the sample to 100 mL with water in a graduated cylinder and place in an acid-washed digestate bottle. If only one graduated cylinder is to be used to volume all samples, the blank is to be the last sample volumed in order to assure against sample carry over. Navy projects require the use of individual graduated cylinders for each sample.

B. If the sample is being prepared for GFAA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and/or Tl, the HCl addition is omitted. The sample is diluted to a final volume of 100 mL in a graduated cylinder, and stored in an acid-washed digestate bottle. If only one graduated cylinder is to be used to volume all samples, the blank is to be the last sample volumed in order to assure against sample carry over.

Note: If a sample is allowed to go to dryness at any stage of the digestion procedure, the sample must be discarded and prepared again.

6.4 If silicates or other insoluble material that could clog the nebulizer or the graphite furnace autosampler are present in the samples, the samples should be allowed to settle overnight prior to analysis. Filtration should be performed only if there is concern that the insoluble material will not settle out of solution. Filter and dilute the samples to a final volume of 100 mL, using acid-washed filter apparatus to avoid sample contamination.

6.5 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain a strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digestates, are recorded on the digestion log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of an empty, acid-washed Erlenmeyer flask to which the appropriate reagents are added and digested in exactly the same manner as a sample The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPS01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately. See Appendix C for instructions on the preparation and use of QC solutions. Spiking levels presented are to be used only as guidance. The actual analytes and concentrations may vary.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in the Appendix C.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP or GF LCSS) must be digested in exactly the same manner as a sample. A Blank Spike consists of an empty, acid-washed Erlenmeyer flask to which the appropriate spikes (determined as outlined in Section 7.3) and reagents are added. The Certified Value and Advisory range for each analyte in a particular LCSS can be accessed from the Quality Control Database.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3050B.

Appendix A. LTL-7015 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.		GRAPHITE FURNACE DIGESTION LOG	Place a √ in the box next to the method performed (circle version)			
SDG #:	Digest start : date/time	Digest finish : date/time	SW-846 3020A		CLP-water	
Analyst:	/	/	SW-846 3050A/B		CLP-soil	

Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments
			Bef	Aft	Bef	Aft			

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	/			

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\METALS\BNCHSHEF\DIG.D

LAUCKS TESTING LABORATORIES, INC.	ICP DIGESTION LOG	Place a √ in the box next to the method performed (circle version).			
SDG #:	Digest start : date/time	Digest finish : date/time	SW-846 3010		CLP-water	
Analyst:	/	/	SW-846 3050A/B		CLP-soil	

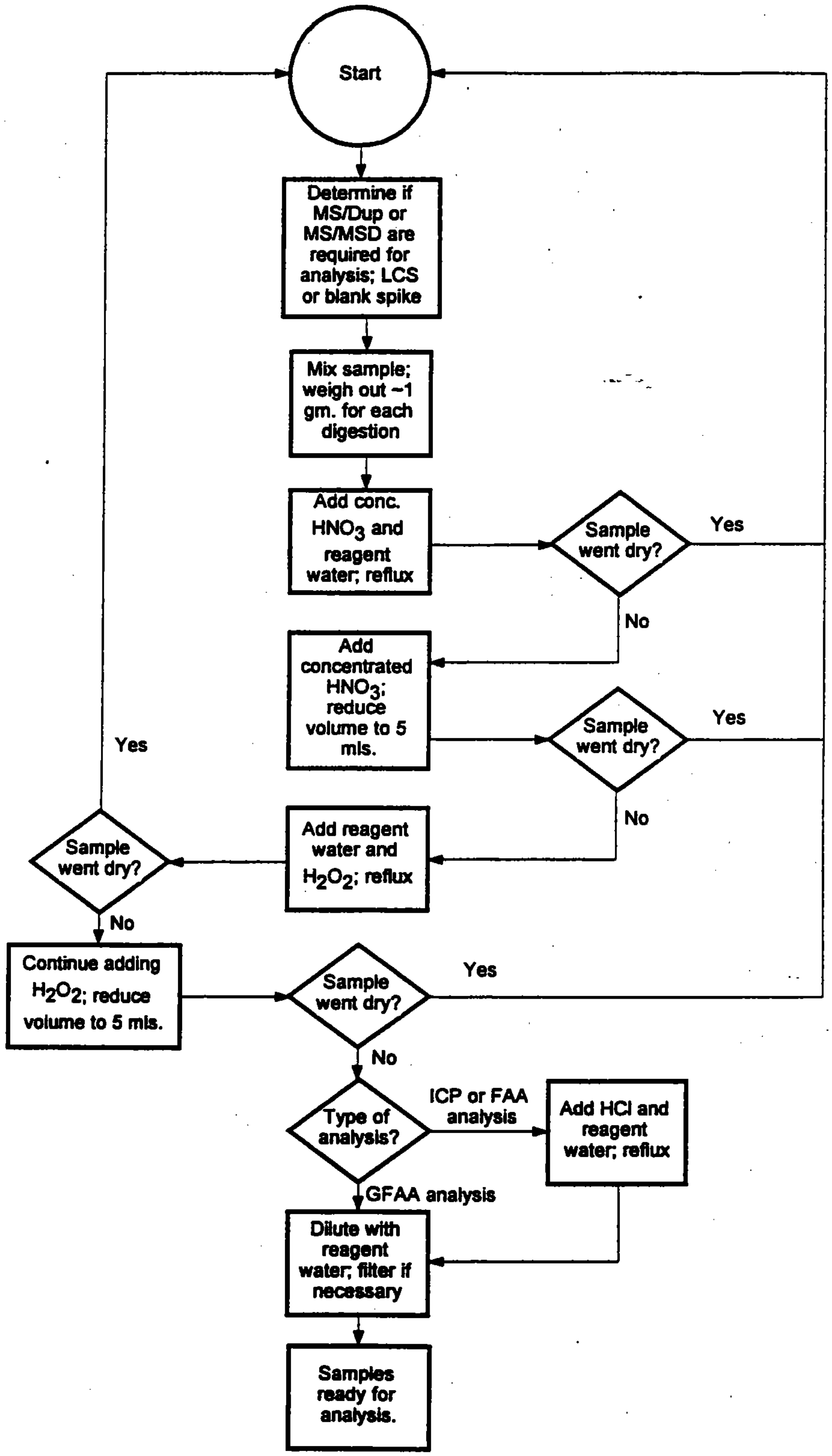
Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color Bef	Color Aft	Clarity Bef	Clarity Aft	Text	Art	Comments

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	/			

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) **Artifacts:** If "Yes", give description in the comments field.

P:\METALS\BNCHSH[illegible]PDIG.D

Appendix B. Flowchart for LTL-7015


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or blank spike
Mix sample; weigh out ~1 gm. for each digestion
Add conc. HNO_3 and reagent water; reflux
Sample went dry?
Yes
No
Add concentrated HNO_3; reduce volume to 5 mls.
Sample went dry?
Yes
No
Add reagent water and H_2O_2; reflux
Sample went dry?
Yes
No
Continue adding H_2O_2; reduce volume to 5 mls.
Sample went dry?
Yes
No
Type of analysis?
ICP or FAA analysis
Add HCl and reagent water; reflux
GFAA analysis
Dilute with reagent water; filter if necessary
Samples ready for analysis.

Appendix C. Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 1000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 100 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 ppm
Pb	200 µL	1000 ppm
Se	100 µL	1000 ppm
Tl	500 µL	1000 ppm

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 ppm	Arsenic
2.0 ppm	Lead
1.0 ppm	Selenium
5.0 ppm	Thallium

- Add 1000 µL of Spike Solution to water samples and 1000 µL to soil samples prior to digestion.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-7101

Title: **Operation of Jarrell-Ash Enviro 36 Simultaneous ICP**

Revision history:

Number	Date
1	01/23/96
2	01/09/97
3	2/4/98

Revised by: Katja Palmer Date: 02/05/98
Katja Palmer, Primary ICP Operator

Approved by: Harry Romberg Date: 2-5-98
Harry Romberg, QA Officer

Approved by: Karen J. Kotz Date: 2/5/98
Karen Kotz, Laboratory Director

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to define the operation of the Jarrell-Ash Enviro 36 (JA36) ICP and any tasks associated with the operation of the instrument. Specific QC requirements and method protocols are listed in their respective SOPs.

2. Equipment List and Standards

◊ Jarrell-Ash Enviro36 simultaneous ICP with autosampler
◊ Calibration standards
◊ Standard reference materials
◊ Coolant circulator
◊ Pump and nebulizer tubing
◊ 13 x 100 mm disposable culture tubes

2.1 Standards

2.1.1 All standards are made in deionized water acidified with 5% HCl and 1% HNO_3. Each completed standard contains 50 ppm of Scandium internal standard. Expiration dates for the standards are based on the expiration date of the purchased stock standard solution. Daily working standards are made up as needed.

2.1.2 The standards used for routine ICP operation are:

◊ A Blank Standard
◊ A High Standard
◊ Mid range standards(for SW846 6010 A only)
◊ CCB (continuing calibration blank)
◊ CCV(continuing calibration verification)
◊ ICB (Initial Calibration blank)
◊ ICV (Initial calibration verification)
◊ ICSA (interference check sample A)
◊ ICSAB (interference check sample B)
◊ CRI (low detection limit sample)

2.1.3 Additionally, every quarter an IDL standard is analyzed, on three non-consecutive days. The exact concentrations of the IDL standards are listed in appendix 2. Annually, the MDL standard is digested and analyzed as part of an MDL study. The concentration of the MDL standard used for the study is documented in the MDL study file.

3. Safetv precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

- Sample digests are acid solutions, use routine acid handling care.
- Concentrated metals standards should be handled with care so that none are absorbed through the skin.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Do not look directly at the torch without the polarizing shield as the UV rays emitted will cause eye damage.

3.1.4 Do not try to operate the instrument with the front door of the torch compartment open by bypassing the interlock.

3.1.5 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.6 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

- Do not attempt to change the power amplifier tube as extremely high voltage may cause electrical shock. Call service engineer to perform this task.
- Do not remove any panels of the RF generator as exposure to microwaves will occur.

3.2 Waste Disposal

3.2.1 Waste is collected in the carboy under the torch compartment. Empty as necessary. Always leave about six-inches of waste in the bottom.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 Calibration Procedure

4.1.1 A SPEX multi-element standard and a Blank standard are used to calibrate the instrument. If running in the autosampler mode, the standards are included at the beginning of the autosampler table.

4.1.2 In the manual mode, select **Operation** from the main menu then select **Analysis.**

4.1.3 Enter the name of the method used or accept the default method. Press F3 for standardization.

4.1.4 Use the arrow keys to highlight the name of the standard. Aspirate the standard and press "F1" (run). Repeat until all standards have been run. Press "F9" twice to return to the analysis menu.

4.2 Quality Control and Limit Check Tables

Note: These tables are method specific (SW846, 200.7 and CLP) and must be applied appropriately.

4.2.1 Quality control check tables are used with QC samples. They contain the true values of the ICVs, the acceptable range of the results according to the method being run, and the units of comparison: percent or absolute. QC Check tables are only used when a sample is defined as a QC sample.

4.2.2 Limit check tables are used with analytical samples and with blank samples (i.e. ICBs and CCBs). The control limits for blanks are dependent on the method being run. They are generally defined as being the absolute value of the result. They should not exceed the reporting limit. For analytical samples their upper range is set as the linear range of the instrument or the level of the high standard, depending on the specifications of the method being run. The lower limit is set at the negative reporting limit, again method specific.

4.2.3 Method information and the QC and Limit tables for each method can be found in **Appendix 3.**

5. Operation procedures

5.1 Startup

- The argon tanks, located in the warehouse, are connected to a delivery manifold that will allow a continuous flow of argon as long as there is 90 psi available in a tank. The line-in pressure regulator is located behind the instrument and is to be adjusted to 50 psi.

- Check pump tubing to see if it is flat or crimped. Change if necessary. Check all connections to see if any blockage exists and if clogs are found, remove them by disconnecting the tubing and flushing with DIW or by using a cleaning wire.

- Turn on control and line breakers on the RF generator.

- Turn on the computer power strip and the power strip attached to the autosampler cart.

- Turn on the torch, auxiliary, and sample argon toggles on the front of the instrument. Let the argon purge through the system for about 3 minutes.

- Settings: torch=18
  aux=0.5
  sample=0.65.

- At the computer select OPERATION, ANALYSIS, accept the default method, and at the command line type **iara** <ENTER>.

- Stretch the rinsate pump tubing into place, set the pressure plate, and turn on the peristaltic pump.

- Prior to igniting the torch aspirate rinse water into the nebulizer. Observe the spray chamber to ensure the aspirated solution is being nebulized.

- Turn off the sample argon toggle to halt gas flow into the nebulizer.

- Press the red RF ON button, then turn the power knob until the forward power reads about 0.5 on the meter.

- Press the ignite button to light the torch. If the torch does not light the first time try again. If it still does not light, adjust the forward power slightly higher until the torch lights while pressing the ignite button. The torch is lit when white streams appear at the top of the torch.

- Slowly increase power until a plasma forms (white "flame"). This will be accompanied by "popping" sounds as the plasma begins to form.

- If the torch appears yellow-orange during this process, depress the blue RF OFF button immediately and turn the power knob back to the off position. This yellow-orange color means that the torch is overheating. See the manufacturers manual, page 14, for corrective action.

- Adjust the forward power to 1.1 KW.

- Switch the automatic power toggle from manual to automatic.

- Slowly turn the power knob fully clockwise.

5.2 Automatic Profiling

- Select OPERATION from the main menu.

- Select ANALYSIS from the menu.

- Accept the default method.

- Set the 5 ppm Mn profiling solution on the autosampler tray at rack #2 position 75 (the white dotted circle). At the command line of the software type **iasrn2srt70ma75** <ENTER>. Translation: Initialize Autosampler, Set Rack Number 2, Set Rack Type 70, Move Autosampler to position #75.

- Select F5 (Profile) from the function keys.

- Select F3 (Automatic) from the function keys.

- Type in a 40 second flush time.

- Press F1 (Run) from the function keys.

- If the peak is between 0.1 and -0.1, the instrument is profiled. If the peak is outside of this range a manual profile is required.

5.3 Manual Profiling

- With the Mn standard still being aspirated, select F1 (manual profile) from the Profile menu.
- Moving the micrometer screw, maximize the reading on the profile meter.
- Move the micrometer screw clockwise until the profile meter reads less than 70% of the maximum. Record the value of the micrometer screw.
- Continue moving the micrometer screw counterclockwise, past the maximum reading on the profile meter, until the profile meter once again reads less than 70% of the maximum. Record the value of the micrometer screw.
- Determine the average of the two values.
- Set the micrometer at the average value by moving the screw in a counterclockwise direction.
- Repeat the auto-profile to check the peak position.

5.4 Analytical Operation

5.4.1 All analytical solutions aspirated into the ICP must contain 50 ppm Sc. 50 µL of 10000 ppm scandium is added to a 10 ml aliquot of sample. If samples are to be run manually aspirate right out of the tube. If they are to be included in an autosampler run, pour the sample into a culture tube and load the tube into the appropriate spot in the autosampler tray.

5.4.2 If samples are to be run in the autosampler mode it is necessary to set up the autosampler table. From the menu choose **Autosampler setup.** Select the table name. Press "F1"(edit autosampler table). Type in the sequence of samples.

5.4.3 To analyze samples in the manual mode it is necessary to be in the Analysis menu. After the instrument has been calibrated, press "F2". Fill in the requested information. Aspirate the ICV. Press "F1"(run). For the ICB, use "F4"(Blank sample). For analytical samples use "F1"(analyze). Repeat for additional reference samples and then for "real" samples.

5.4.4 To analyze samples in the autosampler mode it is also necessary to be in the **Analysis** menu. Enter the desired method or accept the default Method. Calibration will be done by the autosampler table. Press "F9"(Autosampler). Type in the name of the autosampler table and press the "ENTER" key. Verify that the standards and the samples are in the correct positions. Press " F1"(start).

5.4.5 For more details on creating Tables and Methods refer to the Operators Manual for the ICP.

5.5 Shutdown Procedure

5.5.1 Turn the power knob on the RF generator off (left) and press the red reset button when the alarm sounds. Press the blue RF OFF button. Then flip the toggle switch for the automatic power control to the manual position.

5.5.2 The control and line breaker switches only need to be turned off for weekends or if the instrument is not going to be used the next day. This should be done after the RF power has been off for at least 5 minutes.

5.5.3 Turn off the power strip on the autosampler cart.

5.5.4 Release the pressure plates on the peristaltic pump and unhook the pump tubing.

5.5.5 Turn off the torch, auxiliary, and sample argon toggles on the front of the instrument.

5.5.6 If the computer will not be used, turn off the master switch on the computer power strip.

6. Reports

6.1 Results

6.1.1 All Results are organized into Data packets. The format of each packet is dependent on the method and protocol required for that specific job. Details for method specific reports can be found in the method SOP.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the Quality Control database QC_DB. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM (LCSS), blank spike (LCSW) or other report. The recovery values for Ag,Al,Cr,Cd and B in the LCSS and LCSW are plotted on control charts.

6.2.2 Method specific QC requirements are listed in the Method SOP.

7. Quarterly and Yearly Procedures

7.1 Instrument Detection Limits (IDLs)

7.1.1 For complete details on performing IDL studies refer to Laucks SOP LTL-1011. In brief, IDLs for each analyte are determined quarterly by analyzing a "sample" seven consecutive times. This is done on three non-consecutive days. Calculate the standard deviation of the seven results on a given day. Add the standard deviations from the three days and the result is the IDL. The IDL solution is at a concentration of 3-5X the expected IDL. The IDL standard is not digested. The exact concentrations for the IDL standard are listed in **Appendix 2** of the SOP.

7.2 Method Detection Limits (MDLs)

7.2.1 For complete details on performing IDL studies refer to Laucks SOP LTL-1011 . In brief, MDLs for each analyte are determined yearly by analyzing seven replicates of a digested solution. MDLs are run according to the Laucks SOP on MDL determinations using the appropriate method. The MDL solution concentration is documented in the MDL study file.

7.3 Linear Range

7.3.1 CLP and EPA 200.7 - Linear ranges for each analyte are determined by analyzing a high concentration "sample". The analytically determined concentration must be within 5% of the true value. The true value is the upper limit of the ICP linear range. Linear range must be verified quarterly. A list of the ICP linear ranges can be found in **Appendix 2** of this document.

7.3.2 SW846 6010B - The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range limit should be an observed signal with no more than a 10%

variation from the true value of the upper range standard. Linear range must be checked every six months.

7.4 Interelement Correction Factors (IECs)

7.4.1 Interelement correction factors are determined by analyzing single element, high concentration samples. The apparent concentration of an analyte divided by the concentration of an interfering analyte run at 100 ppm, gives the required correction for that analyte/interfering element combination. This must be verified and updated for all analytes and all interfering elements every six months.

Appendix 1

Concentration of analytes in standard solutions in mg/L
for CLP/200.7 and SW846 6010B

ELEMENT	ICAL STD	ICV	CCV	ICSA	ICSAB	CRI
Ag	1.0	0.6	0.5	-	1	0.02
Al	20.	12	10	500	500	-
As	1.0	2.6	2.5	-	1	0.02
Ba	20.	12	10	-	0.5	-
Be	0.5	0.3	0.25	-	0.5	0.01
Ca	50.	300	25	500	500	-
Cd	0.5	0.3	0.25	-	1	0.01
Co	5.0	3	2.5	-	0.5	0.10
Cr	1.0	0.6	0.5	-	0.5	0.02
Cu	2.5	1.5	1.25	200	0.5	0.05
Fe	10.	6	5	-	200	-
Hg	2.0	2	1	-	-	-
K	50.	300	25	500	-	-
Mg	50.	300	25	-	500	-
Mn	1.5	0.9	0.75	-	0.5	0.03
Na	50.	300	25	-	-	-
Ni	4.0	2.4	2	-	1	0.08
Pb	0.5	2.3	1	-	1	0.056
Sb	6.0	3.6	3	-	-	0.12
Se	0.5	2.3	2.5	-	1	0.01
Tl	1.0	0.6	5	-	1	0.02
V	5.0	3	2.5	-	0.5	0.10
Zn	2.0	1.2	1	-	1	0.04
Mo	5.0	10	2.5	-	-	-
B	5.0	10	2.5	-	-	-

* ICV and CCV have different stock solutions

Appendix 1. cont.

Concentration of analytes for SW846 6010A calibration standards in mg/l

ELEMENT	Std 1 (low std)	Std 2 (mid std)	Std 3 (mid-hi std)	Std 4 (high std)
Ag	0.02	0.2	1.0	2.0
Al	1.0	10.	50.	100.
As	0.05	0.5	2.5	5.0
Ba	0.2	1.0	5.0	10.
Be	0.01	0.1	0.5	1.0
Ca	4.0	40	200.	400.
Cd	0.02	0.2	1.0	2.0
Co	0.05	0.5	2.5	5.0
Cr	0.05	0.5	2.5	5.0
Cu	0.05	5.0	25.	50.
Fe	2.0	20.	100.	200
Hg	0.05	0.5	2.5	5.0
K	3.0	30.	150.	300
Mg	2.0	20.	100.	200.
Mn	0.1	1.0	5.0	10.
Na	2.5	25	125.	250.
Ni	0.1	1.0	5.0	10.
Pb	0.1	5.0	25.	50.
Sb	0.05	0.5	2.5	5.0
Se	0.05	0.5	2.5	5.0
Tl	0.1	1.0	5.0	10.
V	0.1	1.0	5.0	10.
Zn	0.1	5.0	25.	50
Mo	0.2	1.0	5.0	10.

* SW846 H.std=ICV

Appendix 2

IDL solutions and linear range for CLP/200.7

ELEMENT	IDL solution T.V. (mg/L)	linear range (mg/L)
Ag	0.01	200.
Al	0.05	600.
As	0.04	300.
Ba	0.01	100
Be	0.002	10.
Ca	0.05	600
Cd	0.005	100
Co	0.01	200
Cr	0.02	200
Cu	0.01	200
Fe	0.05	600
Hg	0.04	-
K	0.45	1000
Mg	0.05	600
Mn	0.02	100
Na	0.05	600
Ni	0.01	200
Pb	0.04	200
Sb	0.02	200
Se	0.05	200
Tl	0.05	10
V	0.04	200
Zn	0.002	200
Mo	0.01	-
B	0.2	-

Appendix 3

Method information, QC and limit check tables

Method: LAUCKIEC

Applicable to CLP/200.7

METHOD INFORMATION

Sample Introduction Device: Normal
Calibration Mode: Concentration

Default Setup:

Number of Repeats : 4
Flush Time (sec) :60.0
Auto-Increment Sample Names?No

Auto-store Analysis Data?	Yes
Auto-store Stdzn Data?	No
Store Individual Repeats?	Yes
Auto-print Analysis Data?	Yes
Auto-print Stdzn Report?	Non
Condensed Print Format?	No

Default File Names:

Analysis Data File	: MIKEDAT
Calibration Data File	: DEFAULT
Calibration Stds Table	: DEFAULT

Autosampler Table	: AUTO
Sample Limits Table	: LINCHK
Blank Limits Table	: MGLBLK
QC Check Table	SPEXCCV

Limit Check Table LINCHK
limit values in ppm

ELEMENT	High limit	Low limit
Ag*	200.	-.01
Al*	600.	-.2
As*	300.	-.3
Ba*	200.	-.1
Be*	100.	-.005
Ca*	600.	-1.
Cd	100.	-.005
Co	200.	-.05
Cr	200.	-.01
Cu	200.	-.025
Fe	600.	-.1
Mg	600.	-1.
Mn	100.	-.015
Na	600.	-1
Ni	200.	-.04
Pb	200.	-.1
Sb	200.	-.06
Se	200.	-.3
Tl	10.	-.5
V	200.	-.05
Zn	200.	-.02
K	1000	-1
B	100.	-.2
Mo	100.	-.01

Limit Check Table MGLBLK
limit values in ppm

Element	High limit	Low limit
Ag*	.01	-.01
Al*	.2	-.2
As*	.2	-.2
Ba*	.2	-.2
Be*	.005	-.005
Ca*	1.	-1.
Cd*	.005	-005
Co*	.05	-.05
Cr*	.01	-.01
Cu*	.025	-.025
Fe*	.1	-.1
Mg*	1.	-1.
Mn*	.015	-.015
Na*	1.	-1.
Ni*	.04	-.04
Pb*	.05	-.05
Sb*	.06	-.06
Se*	.2	-.2
Tl*	.5	-.5
V*	.05	-.05
Zn*	.02	-.02
K*	1.	-1.

QC Check Table SPEXICVM and BMOICVM
limit values in ppm

Element	Value	% Range
Ag*	.6	10
Al*	12.	10
As*	2.6	10
Ba*	12.	10
Be*	.3	10
Ca*	300.	10
Cd*	.3	10
Co*	3	10
Cr*	.6	10
Cu*	1.5	10
Fe*	6.	10
Mg*	300.	10
Mn*	.9	10
Ni*	2.4	10
Pb*	2.3	10
Sb*	3.6	10
Se*	2.3	10
Tl*	.6	10
V*	3.	10
Zn*	1.2	10
K*	300.	10
B	10	10
Mo	10	10

QC Check Table SPEXCCVM
limit values in ppm

Element	Value	% Range
Ag*	.5	10
Al*	10	10
As*	2.5	10
Ba*	10	10
Be*	.25	10
Ca*	25	10
Cd*	.25	10
Co*	2.5	10
Cr*	.5	10
Cu*	1.25	10
Fe*	5	10
Mg*	25	10
Mn*	.75	10
Na*	25	10
Ni*	2	10
Pb*	1	10
Sb*	3	10
Se*	2.5	10
Tl*	5	10
V*	2.5	10
Zn*	1	10
K*	25	10
Hg*	1	10
B*	2.5	10
Mo*	2.5	10

* QC Check Table SPEXCCVM valid for CCVs in all current Methods

Method: SW846

Applicable to SW846 Soils

METHOD INFORMATION

Sample Introduction Device: Normal
Calibration Mode: Concentration

Default Setup:

Number of Repeats : 4
Flush Time (sec) : 60.0
Auto-Increment Sample Names? No

Auto-store Analysis Data? Yes
Auto-store Stdzn Data'? No
Store Individual Repeats? Yes
Auto-print Analysis Data? Yes
Auto-print Stdzn Report : None
Condensed Print Format? No

Default File Names:

Analysis Data File : MIKEDAT
Calibration Data File : DEFAULT
Calibration Stds Table: DEFAULT

Autosampler Table : AUTO
Sample Limits Table : HCALSTD
Blank Limits Table : MDL2X
QC Check Table : SPEXCCVM

Limit Check Table HICALSTD
limit values in ppm

Element	High limit	Low Limit
Ag*	2	-.0055
Al*	100	-.221
As*	5	-.057
Ba*	10	-.038
Be*	1	-.003
Ca*	400	-1.008
Cd*	2	-0063
Co*	5	-.0129
Cr*	5	-.0199
Cu*	50	-.0177
Fe*	200	-.4988
Mg*	200	-.5198
Mn*	10	-.0247
Na*	250	-.5528
Ni*	10	-.0365
Pb*	50	-.059
Sb*	5	-.033
Se*	5	-.1005
Tl*	10	-.0668
V*	10	-.0255
Zn*	50	-.036
K*	300	-.5472
MO*	10	-0783

* HCALSTD Table low limit based on 2X current soil MDL and subject to change annually

Limit Check Table MDL2X
limit values in ppm

Element	High Limit	Low Limit
Ag*	.0055	-.0055
Al*	.221	-.221
As*	.057	-.057
Ba*	.038	-.038
Be*	.003	-.003
Ca*	1.008	-1.008
Cd*	.0063	-.0063
Co*	.0129	-.0129
Cr	.0199	-.0199
Cu	.0177	-.0177
Fe*	.4988	-.4988
Mg*	.5198	-.5198
Mn*	.0247	-.0247
Na*	.5528	-.5528
Ni*	.0365	-.0365
Pb*	.059	-.059
Sb*	.033	-.033
Se*	.1005	-.1005
Tl*	.0668	-.0668
V*	.0255	-.0255
Zn*	.036	-.036
K*	.5472	-.5472
Mo*	.0783	-.0783

* Blank Limits based on 2X soil MDL and therefore subject to annual variations

QC Check Table **HSTDICVM**

limit values in ppm

Element	Value	% Range
Ag*	2	5
As*	5	5
Ba*	10	5
Ca*	400	5
Be*	1	5
Al*	100	5
Cd*	2	5
Co*	5	5
Cr*	5	5
Cu*	50	5
Fe*	200	5
Hg*	5	5
K*	300	5
Mg*	200	5
Mn*	10	5
Na*	250	5
Ni*	10	5
Pb*	50	5
Sb*	5	5
Se*	5	5
Tl*	10	5
V	10	5
Zn*	50	5
Mo*	10	5

Method: SW846W

Applicable to SW846 Waters

METHOD INFORMATION

Sample Introduction Device: Normal
Calibration Mode: Concentration

Default Setup:

Number of Repeats	4	Auto-store Analysis Data?	Yes
Flush Time (sec)	60.0	Auto-store Stdzn Data?	No
Auto-Increment Sample Names?	No	Store Individual Repeats?	Yes
		Auto-print Analysis Data?	Yes
		Auto-print Stdzn Report :	None
		Condensed Print Format?	No

Default File Names:

Analysis Data File:	MIKEDAT	Autosampler Table	AUTO
Calibration Data File:	DEFAULT	Sample Limits Table	HCALSTDW
Calibration Stds Table:	DEFAULT	Blank Limits Table	WMDL2X
Calibration Stds Table:	DEFAULT	QC Check Table	SPEXCCVM

Limit Check Table HCALSTDW
limit values in ppm

Element	High Limit	Low Limit
Ag*	2	-.0082
Al*	100	-.1864
As*	5	-.0528
Ba*	10	-.0119
Be*	1	-.001
Ca*	400	-.346
Cd*	2	-.0213
Co*	5	-.0057
Cr *	5	-.0157
Cu*	50	-.0051
Fe*	200	-.163
Mg*	200	-.253
Mn*	10	-.008
Na*	250	-.1975
Ni*	10	-.0086
Pb*	50	-.04
Sb*	5	-.0154
Se*	5	-.0698
Tl	10	-.071
V*	10	-.0051
Zn*	50	-.0147
K*	300	-.288
Mo*	10	-.0233

* Low sample limit bases on 2X Water MDL. Subject to change annually.

Limit Check Table WMDL2X
limit values in ppm

Element	High Limit	Low Limit
Ag*	.0082	-.0082
Al*	.1864	-.1864
As*	.0528	-.0528
Ba*	.0119	-.0119
Be*	.0009	-.0009
Ca*	.346	-.346
Cd*	.0213	-.0213
Co*	.0057	-.0057
Cr*	.0157	-.0157
Cu*	.0051	.0051
Fe*	.163	-.163
Mg*	.253	-.253
Mn*	.008	-.008
Na*	.1975	-.1975
Ni*	.0086	.0086
Pb*	.04	-.04
Sb*	.0154	-.0154
Se*	.0698	-.0698
Tl*	.071	-.071
V*	.0051	-.0051
Zn*	.0147	-.0147
K*	.288	-.288
Mo*	.0233	-.0233

* Blank limits bases on current Water MDLs. Subject to annual variation.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7105

Title: Method Protocols for the Analysis of Metals by ICP Using SW 846 6010B

Revision history:

Number	Date
0	1/29/98

Written by: Katja Palmer
Katja Palmer, ICP operator
Date: 01/29/98

Reviewed by:
Ming-Hwa Hwang, Metal Supervisor
Date: 1/29/98

Approved by: Harry Romberg
Harry Romberg, Quality Assurance Officer
Date: 1-29-98

Approved by: Karen Kotz
Karen Kotz, Laboratory Director
Date: 1/29/98

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1 Scope

This method is to be used as a supplement to the instrumental SOP in order to follow the method requirements of SW 846 6010B for ICP analysis. Operating parameters are to be followed in the individual instrument SOP.

The analyst should become familiar with SW 846 protocols prior to using this SOP.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

Samples are to be collected in either glass or plastic containers. Water samples are to be preserved to a pH < 2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C. At least 200 grams of sample should be collected. The holding time for ICP metals is 6 months. See Appendix III for Sample Handling and Preservation Table.

1.3 Definition of Terms

This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

ICV - Initial Calibration verification - This is a standard run immediately following the initial calibration. The ICV is made from an independent source. Agreement within 10% and a relative standard deviation less than 5% RSD from replicate (minimum of two) integrations, is required.

ICB - Initial calibration blank - An instrument blank is made up in the same way as calibration standards, without target analytes.

CCV - Continuing calibration verification. - This is a standard analyzed after every 10 samples during the analysis sequence to determine whether the instrument or system has remained in calibration. Agreement within 10% and less than 5% RSD from replicate (minimum of two) integrations, is required.

CCB - Continuing Calibration Blank - This is a blank that is used to determine if there is carry-over between sample injections. A CCB succeeds every CCV, every 10 samples.

ICSA - Interference Check Solution A - This solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.

ICSAB - Interference Check Solution AB - This solution is prepared by spiking the ICSA with known quantities of analyte. Adequate recovery of the analytes within this interfering matrix indicates proper application of the correction factors.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

IDL - Instrument detection limit - The lowest concentration of a target analyte that is detectable. The IDL is three times the average standard deviation of seven replicates at 2 to 5 times the estimated IDL over three non consecutive days. Used as a starting point for selecting MDL study spiking levels. IDL should be determined quarterly.

MDL - Method detection limit - The lowest concentration which will yield a positive result that is greater than zero at a known level of confidence. The MDL is preparation specific and empirically determined by Laucks.

LCS - Laboratory Control Sample - This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis.

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

Sequence - A set of sample digests and standard solutions introduced into an instrument in a chronologically continuous group.

2. Equipment List and Standards

2.1 Instrumentation:

Thermo Jarrell Ash Enviro 36 simultaneous ICP or equivalent.

2.2 Standards

SW 846 requires the use of one standard and a blank. See the instrument SOP for standards and their preparation.

3. Safety precautions and Waste Disposal

See instrument SOP for detailed safety precautions and waste disposal.

Safety

All standards, samples and sample solutions should be handled as if they are hazardous substances.

Refer to the instrument manufacturer's manual for routine instrument precautions.

Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

Caution should be used when handling acidic digestates.

3.1 Waste Disposal

Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range limit should be an observed signal with no more than a 10% variation from the true value of the upper range standard. All samples with elements exceeding the level of the upper range standard must be diluted.

4.3 Initial Calibration

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration and a 5% RSD for replicate integrations.

Criteria and Corrective Action:

Since a linear regression is not possible when using a two point calibration on the Enviro 36, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed..

4.4 Initial Calibration Standard ICV:

Immediately after the calibration curve, an ICV is analyzed.Criteria:

The calculated concentration of the standard must be within 10% of the expected value and the RSD must be less than 5% for replicate integrations.

Corrective Action:

If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.5 Initial Calibration Blank

After the analysis of the high standard, an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed three times the instrument detection limit.

Corrective Action:

If the ICB analyte levels exceed 3 times the IDL, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the backgroundl mean, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.6 Continuing Calibration Verification (CCV) and Blank (CCB)

A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run. The CCV must also be prepared from an independent source.

The CCV must fall within ± 10% of the true value and the RSD must be less than 5% for replicate integrations.

The levels of target analytes in the CCB should not exceed 3 times the IDL.

Corrective action:

If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV/CCB which is in control or by preceding and following CCV/CCBs which are within limits.

If the CCB analyte levels exceed 3 times the IDL, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be preformed before continuation of the run.

4.7 Interference Check Solutions A (ICSA) and AB (ICSAB)

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interefering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the CRDL.

Corrective Action:
If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.8 Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the MDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) The reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interference caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit. a narrative comment must be made which documents the corrective actions taken.

4.9 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limit are 80% to 120%. Control limits for the LCSW will be empirically determined and must be within the method specified control limit noted aboved.

LCSS(soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits, then a redigestion will occur for the affected analytes.

4.10 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$recovery,\% = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spike added, the amount of spiking material actually added calculated on the sample basis.

For ICP, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance. The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In some cases a Post Digestion Spike is required when matrix interference is suspected. In all cases a narrative explanation of the condition is required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event..

4.11 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

At least one matrix spike duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|SS - SSD|}{(SS + SSD)/2} * 100$$

where:

SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

For sample concentractions greater than ten times the IDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than ten times the IDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control RPD will be flagged with an "*".

4.12 Serial Dilution

A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. If the difference is greater than 10% on analytes that exhibit a level 50 times greater than the MDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective action is necessary other than appropriately flagging the data.

5. Operation procedures

- Calibrate the instrument with a blank and one standard.

- Immediately following the calibration, analyze the ICV followed by an ICB. Concentration values obtained for the ICV should not deviate from the actual values by more than 10% and less than 5% RSD for replicate integrations.
- Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample.
- Analyze a CCV and CCB every 10 samples. The CCV must agree within 10% of its expected value and less than 5% RSD for replicate integrations. The results of the calibration blanks are to be within 3 times the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within 3 times the IDL, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples.

5.1 Analysis Sequence

See Appendix IV for Analytical Sequence

5.2 Instrumental Conditions

See instrument SOP for operating procedures.

5.3 Analytical Operation

See instrument SOP for operating procedures.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines can be reported via CLP SOW 3/90 forms. These forms provide all relevant QC information.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the MDL and the reporting limit.	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁
E	The Serial Dilution did not agree within 10%.	❁
S	The analyte concentration was determined by MSA.	❁

◆ Used in all reports.
❁ Used in data validatable packages.

6.2 Control Chart(s)

The recovery values for ICP analytes Al, B, Cd, Cr, and Ag in the LCS are plotted on control charts. Other analytes may be added at the discretion of QA without immediate revision of this SOP.

6.3 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6010B, Revision 2, December 1996

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions

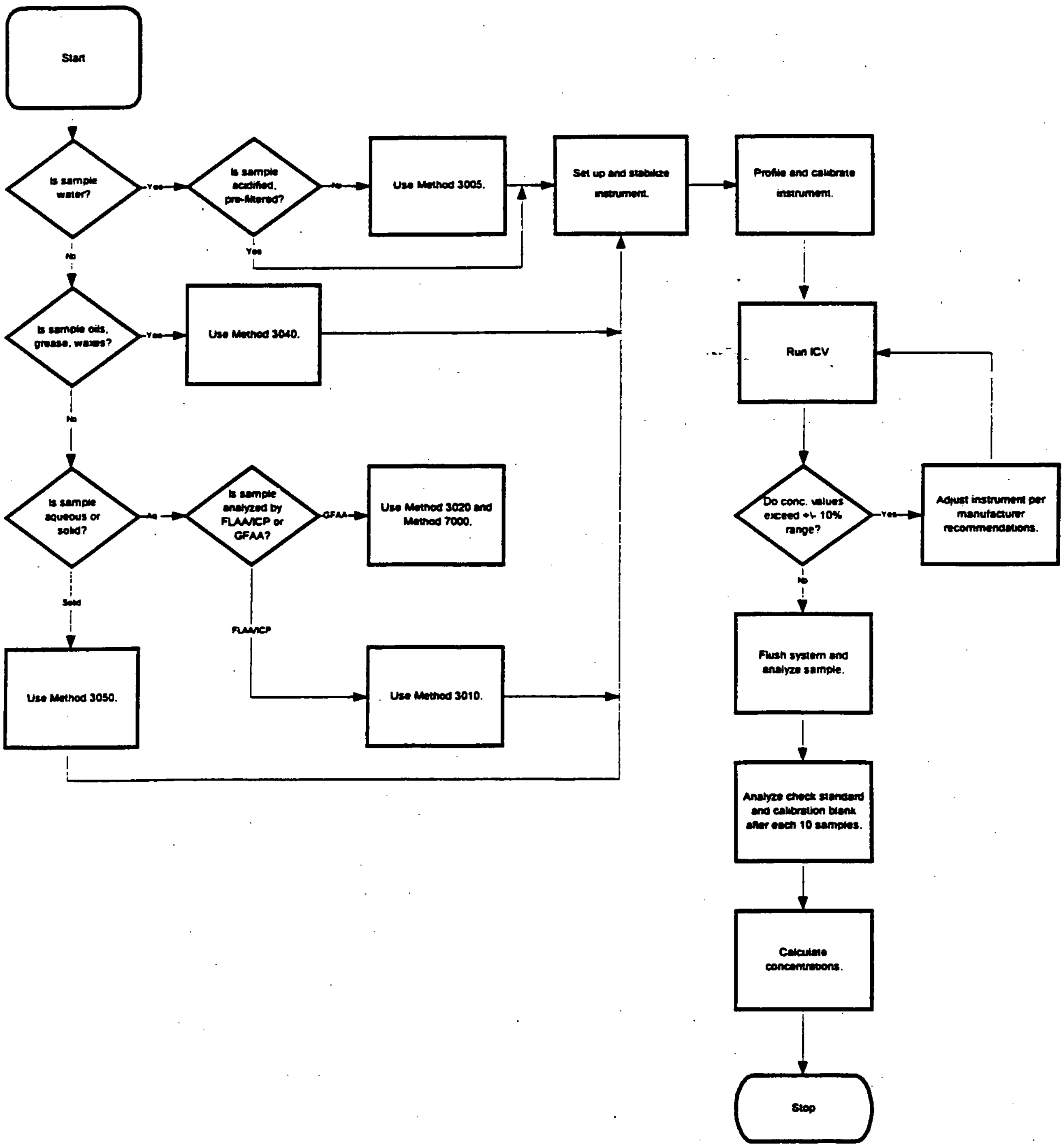
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documenta
Initial Calibration	One standard and a Blank	One standard and a Blank	Beginning of each run.		In raw data.
Initial Calibration Verification	±10% of nominal value. Made from an independant source.	±10% of nominal value. Made from an independant source.	Beginning of each run immediately following ICAL.	Recalibration required.	Form 2A or raw data.
Continuing Calibration Verification	±10% and RSD < 5% for replicate integrations.	±10% and RSD < 5% for replicate integrations.	Every 10 samples. Mid-range	Recalibrate and rerun affected samples.	Form 2A or raw data.
Instrument Blank	±3 sigma of average.	±3 sigma of average	Every 10 samples.	Recalibrate and rerun affected samples.	Form 3 or i raw data.
Method Blank	< MDL	< reporting limit	One/batch	Redigest samples	Form 3, in r data, or database re
Laboratory Control Sample		LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples	Form 7, in data, or database re
Matrix Spike Recovery	75-125%	75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5, in data, or database re
Duplicate % Difference	± 20%	± 20% or current QC database criteria.	5% or per batch	Consult QCO	Form 6, in data, or database re
Serial Dilution	± 10% difference	± 10% difference	One/batch	Flag data with an "E".	Form 9 or raw data.
ICSA,AB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	Consult QCO	Form 4, or raw data.

Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentat**
Standard Reference Material (SRM) Recovery	See QC control catalog	Control limits set by vendor	5% or per batch	Redigest samples	Form 7, in r data, or database rep

Appendix II

Flow Chart


Start
Is sample water?
Yes
No
Is sample acidified, pre-filtered?
No
Yes
Use Method 3005.
Set up and stabilize instrument.
Profile and calibrate instrument.
Is sample oils, grease, waxes?
Yes
No
Use Method 3040.
Run ICV
Is sample aqueous or solid?
Aq
Solid
Is sample analyzed by FLAA/ICP or GFAA?
GFAA
FLAA/ICP
Use Method 3020 and Method 7000.
Do conc. values exceed +/- 10% range?
Yes
No
Adjust instrument per manufacturer recommendations.
Use Method 3050.
Use Method 3010.
Flush system and analyze sample.
Analyze check standard and calibration blank after each 10 samples.
Calculate concentrations.
Stop

Appendix III

Sample Handling and Preservation

Metals (except hexavalent chromium and mercury):

Measurement	Digestion Volume Required.	Collection Volume	Preservation/Holding Time
Total recoverable	100 mL	600 mL	HNO_3 to pH <2 6 months
Dissolved	100 mL	600 mL	Filter on site; HNO_3 to pH <2 6 months
Suspended	100 mL	600 mL	Filter on site 6 months
Total	100 mL	600 mL	HNO_3 to pH <2 6 months

Solid samples should be at least 200 g and usually require no preservation other than storing at 4°C until analyzed. Either plastic or glass containers may be used for sample collection.

Appendix IV

Analytical Sequence

Standard 0
Standard 1
ICV
ICB
CCV
CCB
ICSAI
ICSABI
Sample #1
Sample #1D
Sample #1S
Sample #1L
Sample #2
Sample #3
Sample #4
ICSAF
ICSABF
CCV
CCB

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7202

Title: Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS), SW 846 Method 6020

Revision history:

Number	Date
1.0	04/26/95
2.0	02/09/98
3.0	4/22/98
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Written by: ________________ Date: 7-22-99
Ming-Hwa Hwang, Metals Supervisor

Approved by: Harry Romberg Date: 7-22-99
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 7-22-99
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 **Assigned to:** Tetra

Table of Contents

1. Introduction and Scope

1.1 Method Description

Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique which is applicable to µg/L concentrations of a large number of elements in water and wastes after appropriate sample preparation steps are taken. When dissolved constituents are required, samples must be filtered and acid preserved prior to analysis. No further digestion is required prior to analysis for dissolved elements. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soil, sludge, sediment, and other solid waste for which total (acid-leachable) elements are required.

See Appendix V for reporting limits.
See Appendix I for analytical masses and standard concentrations.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated their ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

Water samples should to be collected in plastic or Teflon containers and preserved to a pH < 2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C and may be collected in glass if plastic containers are not available. At least 200 grams of sample should be collected. The holding time for metals is 6 months. If mercury is being analyzed by this technique, which is not currently approved or done without specific client arrangement, the holding time is 28 days.

1.3 Definition of Terms

This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier - A number given to each analysis group which uniquely identifies that batch. This number is preceded by an "A", mmddyy, ICPMS, matrix (W for water, S for soil), sequence number (i.e. A022595ICPMSW01).

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same matrix as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate.

IDL - Instrument detection limit. IDL's can be estimated by analyzing seven replicates of a standard analyte solution over three nonconsecutive days. The analyte concentration should be 3-5 times the estimated IDL. Multiplying the average standard deviation by three will yield the IDL for that analyte. Each measurement must be performed as though it were a separate analytical sample. IDL's must be determined quarterly

MDL - Method detection limit - The lowest concentration of an analyte which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined and are performed annually.

LCS - Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

Serial Dilution - If the analyte concentration is within the linear range of the instrument and is sufficiently high (minimally, a factor of 100 above the IDL/MDL), an analysis of a fivefold dilution must agree within ±10% of the original determination. If not, an interference effect must be suspected. One serial dilution must be analyzed for each twenty samples or less of each matrix in a batch. A serial dilution is denoted in the raw data by an "L".

ICSA - Interference Check Solution A. The ICSA is a solution that contains the interfering analytes. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the analytes of interest.

ICSAB - Interference Check Solution AB. The ICSAB is a solution that contains the interfering analytes and the analytes of interest. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the recovery of the analytes of interest.

Internal Standards - Internal standards are added to all blanks, standards, and samples. They monitor the affect of a sample's matrix on the quantification of the analytes of interest. The internal standards used are Sc45, In115, and Bi209.

Post-Digestion Spike - An analyte spike added to a portion of a prepared sample should be recovered to within 75% to 125% of the known value or within the laboratory derived acceptance criteria.

Standard-Addition - The standard addition technique involves adding known amounts of standard to an aliquot of the sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope than that of the calibration standards.

QC Period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

2. Equipment List and Standards

2.1 Instrumentation:

Perkin-Elmer ELAN 5000

2.2 Standards

SW 846 requires the use of one standard and a blank. Standards are made in a 1% HNO_3. See Appendix I for standard concentrations.

2.3 Internal Standards

Sc45, In115 and Bi209 are used as internal standards.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

All standards, samples and sample solutions should be handled as if they are hazardous substances.

Refer to the instrument manufacturer's manual for routine instrument precautions.

Routine precautions include an awareness of the moving parts on the instrument you are using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

Radio Frequency - The ELAN 5000 uses a high energy RF. Although the generator is well shielded, care should be taken when operating the instrument. Pace makers can be adversely affected by exposure to high energy RF.

Plasma Radiation - Do not view the torch without the proper eye wear. Severe eye damage can occur if the plasma is viewed directly.

3.2 Waste Disposal

Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal.

4. Quality Control

4.1 Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

Linear ranges for each analyte are determined by analyzing a high concentration "sample". The analytically determined concentration must be within 5% of the true value. The true value is the upper limit of the ICP/MS linear range. Linear ranges must be verified quarterly.

4.3 Internal Standards

A 50 uL aliquot of a 20 ppm of Sc45, 10 ppm of In115 and 10 ppm of Bi209 is added to a 10 mL of all standards and samples prior to analysis.

Criteria

The intensities of all internal standards for instrument check standards must be between 80 and 120 percent of the intensities of the internal standards in the initial calibration standard.

Corrective action

If the criteria are not met, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and re-analyze the affected samples.

Criteria

The intensities of all internal standards in the samples must be between 30 and 120 percent of the intensities of the internal standards in the initial calibration standard.

Corrective action

When the intensity of any internal standard in the sample fails to fall between the required levels, the sample must be diluted fivefold (1+4) and re-analyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until all internal standards fall within the prescribed windows.

4.4 Initial Calibration

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration.

Criteria and Corrective Action:

Since a linear regression is not possible when using a two point calibration on the ELAN 5000, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed..

4.5 Initial Calibration Verification

Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

Criteria

The calculated concentration of the ICV should be within 90%-110% of the true value.

Corrective action

If the ICV criteria are not met, the analysis is terminated. Perform system maintenance and re-calibrate the instrument.

4.6 Initial Calibration Blank

After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed the contract required detection limit .

Corrective action

If the initial ICB contains target analyte levels above the contract required detection limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.7 Continuing Calibration Verification (CCV) and Blank (CCB)

A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

Criteria

The CCV must fall within ± 10% of the true value.

The levels of target analytes in the CCB should not exceed the contract required detection limit.

Corrective action

If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

If the initial CCB contains target analyte levels above the contract required detection limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.8 Interference Check Solutions A (ICSA) and AB (ICSAB)

Due to the high sensitivity of the ICP-MS technique and instrument developments that have occurred since the method was written, the high dissolved solids content of the specified ICS solutions are not recommended by the manufacturer for modern instruments. The ICSA and ICSAB solutions are prepared at different concentration levels from method 6020 to avoid clogging of the sampler cone orifice and damage to the instrument. Therefore, Al, Ca, Fe, Mg, Na, P, K, S, C, and Cl in the ICSA and the ICSAB are at 1/10 of the specified levels. See Appendix II for ICSA and ICSAB solutions concentrations.

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interfering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the contract required detection limit.

Corrective Action:
If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.9 Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the CRDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) The reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interference caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.10 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limits are 80% to 120%.

LCSS(soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits, a redigestion will occur for the affected analytes.

4.11 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$recovery,\% = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spike added, the amount of spiking material actually added calculated on the sample basis.

For ICP/MS, control limits for spike recoveries will be 75-125% unless the sample result is greater than 4 times the spike concentration or unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance.
The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event.

4.12 Post-Digestion Spike

A post digestion spike is also performed to a portion of a prepared sample. The minimum frequency for MS analysis is 1 each per 20 samples per matrix, control limits for post-spike recoveries will be 75-125%

Corrective Action:

Samples with post-spike recoveries outside control limits will be diluted and re-analyzed to compensate for matrix effects. The results must agree to within 10% of the original measured concentrations. A standard-addition technique may also be used to compensate for matrix effects.

4.13 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

At least one matrix spike duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|SS - SSD|}{(SS + SSD)/2} * 100$$

where:
SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

For sample concentrations greater than 5 times the CRDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than 5 times the CRDL, control limits for the difference will be ± the CRDL. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control RPD will be flagged with an "*".

4.14 Serial Dilution

A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. If the difference is greater than 10% on analytes that exhibit a level 50 times greater than the IDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective is necessary other than appropriately flagging the data.

5. Procedure

5.1 Instrumental Conditions

- Refer to manufacture's instruction for specific operating procedures. Allow at least 30 minutes for the instrument to stabilize before initiating any analysis.

- Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria which must be met prior to any sample being analyzed. The monitored masses of Mg, Rh, and Pb must meet the following criteria:

Element	RSD for replicate (minimum of four) integrations.	Mass, amu	Resolution @ 10 % peak height, amu
Mg	< 5%	23.90-24.10	<0.9
Rh	< 5%	102.80-103.00	<0.9
Pb	< 5%	207.90-208.10	<0.9

5.2 Analytical Operation

- Calibrate the instrument, using a calibration blank and a standard. Refer to Appendix I for the applied levels of concentration.

- All masses which could affect data quality are monitored to determine potential effects from matrix components on the analytes of interest.

- After the calibration has been established, an ICV solution is analyzed to verify the validity of the curve. Measurements for the analytes of interest must be at ±10% of the true value. A re-calibration and re-analysis is required for any analyte which falls outside the control limit.

- Analyze the interference check samples (ICSA and ICSAB) prior to and after the analysis of samples

- Analyze a CCV and a CCB once every 10 analytical samples.

- Dilute samples that exceed the established linear range of the instrument.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines the data can be reported via CLP SOW 3/90 forms.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the MDL and the reporting limit.	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁
E	The Serial Dilution did not agree within 10%.	❁
S	The analyte concentration was determined by MSA.	❁

◆ Used in all reports.
❁ Used in data validatable packages.

6.2 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6020, Revision 0, September 1994

Appendix I

Standard Solutions

Elements	Mass	STD, µg/L
Be	9	50
Na	23	500
Mg	26	500
Al	27	400
K	39	500
Ca	44	500
V	51	100
Cr	52	100
Mn	55	150
Fe	54,57	200
Co	59	100
Ni	60	400
Cu	63,65	50
Zn	66	200
As	75	100
Se	82	200
Ag	107	100
Cd	111	50
Sb	121	60
Ba	137	400
Tl	205	100
Pb	208	50
Li	7	200
B	11	200
Mo	98	200
Sn	118,120	200
U	238	250

The STD is made by diluting 10.0 mL of the ICP/MS stock standard to a 100 mL final volume.

The ICP/MS stock standard which consists of:

100 µL	ICAL-1
1000 µL	ICAL-2
200 µL	ICAL-3
1000 µL	ICAL-4
100 µL	ICAL-5
250 µL	1000 ppm U
200 µL	1000 ppm B
200 µL	1000 ppm Mo
200 µL	1000 ppm Li
200 µL	1000 ppm Sn
40 µL	1000 ppm Be
150 µL	1000 ppm Se

Note: 50 µL of 20 ppm Sc45, 10 ppm In115 and 10 ppm Bi209 is added to a 10 mL aliquot of standard.

Appendix II

ICSA and ICSAB Solutions

Element	ICSA (PPM)	ICSAB (PPM)
Aluminum	4	4
Arsenic		0.020
Cadmium		0.020
Calcium	12	12
Chromium		0.040
Cobalt		0.040
Copper		0.040
Iron	10	10
Magnesium	4	4
Manganese		0.040
Molybdenum		0.16
Nickel		0.040
Potassium	4	4
Selenium		0.020
Silver		0.040
Sodium	10	10
Vanadium		0.040
Zinc		0.020

Appendix III

QC Summary Table

Laucks Testing Laboratories

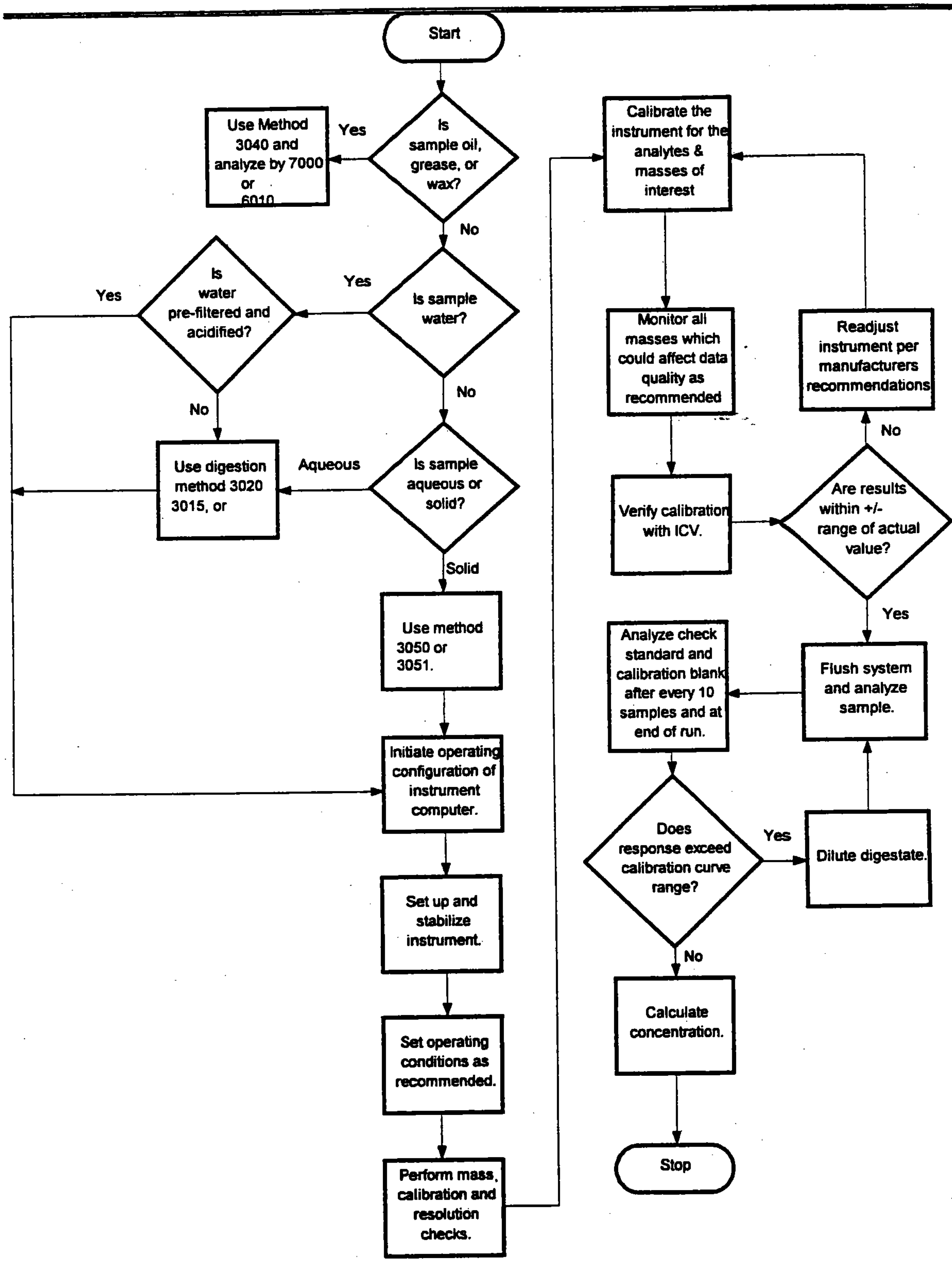
Method SW 846 6020 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Mass Calibration Check	must be performed in mass regions of interest and be within ±0.1 amu of the actual value.	Mg 23.90-24.10 Rh 102.80-103.00 Pb 207.90-208.10	Beginning of each analysis.	Perform new mass calibration.	Instrument Logbook
Resolution Check	< 0.9 amu full width at 10% peak height	< 0.9 amu full width at 10% peak height	Beginning of each analysis.	Adjust resolution.	Instrument Logbook
Initial Calibration	Blank and at least one standard	Blank and one standard	Beginning of each analysis	NA	In the raw data and/or on FORM 14.
Initial Calibration Verification	±10% of true value. Made from an independent source.	±10% of true value. Made from an independent source..	Immediately following calibration.	Recalibrate and reverify.	Form 2, in the raw data
Initial Calibration Blank	Values must be < 3x the IDL for each element.	Values must be < CRDL	Immediately following ICV.	Recalibrate, reverify, and rerun the ICB.	Form3, in the raw data
Continuing Calibration Verification	±10% true value.	±10% true value. Analyte levels are at the mid-range of the calibration.	Every 10 samples and end of run.	Recalibrate and rerun affected samples.	Form 2, in the raw data
Continuing Calibration Blank	Values must be < 3 times the IDL for each element.	Values must be < CRDL	Immediately following CCV.	Recalibrate and rerun affected samples.	Form 3, in the raw data
Method Blank	< CRDL or <5% of regulatory limit or any sample	< CRDL or <5% of regulatory limit or any sample	One/batch	Redigest samples	Form 3, in the raw data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Internal Standards	1.Samples: 30 % - 120 % of the initial calibration standard 2. Instrument Check Standards: 80 -120 of the initial calibration standard.	1. Samples 30 % - 120 % of the initial calibration blank. 2. Instrument Check Standards: 80 -120 of the initial calibration blank.	1. All samples 2. All instrument check standards	1. dilute and reanalyze. 2. recalibrate, re-analyze the affected samples.	In the raw data.
Serial Dilution	within ±10% of the original value if the analyte conc. is > 100 x the IDL.	within ±10% of the original value if the analyte conc. is > 100 x the IDL.	One/batch	Flag data with an "E".	Form 9,.in the raw data
Duplicate, % Difference	± 20% for analyte values greater than 100 times the IDL.	± 20% or current QC database criteria.	5% or per batch	reanalyze digestates, if still fail, consult QCO	Form 6, in raw data, or database report
Matrix Spike Recovery		75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5A, in raw data, database report
Post-Digestion Spike	75%-125%	75%-125%	5% or per batch	dilute and re-analyze or MSA	Form 5B, in the raw data
Laboratory Control Sample	One/batch, no acceptance criteria	LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples.	Form 7, in raw data, or database report
ICSA and ICSAB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	reanalyze affected samples	Form 4, or in raw data.

Appendix IV

Flow Chart


Start
Is sample oil, grease, or wax?
Yes
Use Method 3040 and analyze by 7000 or 6010
No
Is sample water?
Yes
Is water pre-filtered and acidified?
Yes
No
Use digestion method 3020 3015, or
Aqueous
Is sample aqueous or solid?
No
Solid
Use method 3050 or 3051.
Initiate operating configuration of instrument computer.
Set up and stabilize instrument.
Set operating conditions as recommended.
Perform mass, calibration and resolution checks.
Calibrate the instrument for the analytes & masses of interest
Monitor all masses which could affect data quality as recommended
Verify calibration with ICV.
Are results within +/- range of actual value?
No
Readjust instrument per manufacturers recommendations
Yes
Flush system and analyze sample.
Analyze check standard and calibration blank after every 10 samples and at end of run.
Does response exceed calibration curve range?
Yes
Dilute digestate.
No
Calculate concentration.
Stop

Appendix V

Routine Reporting Limits

SW 846 6020 Reporting Limits

Element	Reporting Limit, µg/L	Element	Reporting Limit, µg/L
Ag	3.0	Mn	1.0
As	1.0	Mo	10.0
B	10.0	Ni	10.0
Ba	2.0	Pb	1.0
Be	1.0	Sb	1.0
Cd	1.0	Se	3.0
Co	3.0	Sn	10.0
Cr	5.0	U	100
Cu	2.0	V	2.0
Li	10.0	Zn	10.0

Reporting limits are approximately 2-10 times the instrumental MDL. The MDL is based on samples prepared using SW 846 3015. Values actually reported may be less than the routine reporting limits but above our method detection limit.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8000

Title: **Determination of Retention Time Windows**

Revision history:

Number	Date
0	10/30/94
1	1/30/98

Written by: ______________________ Date: 2/2/98
Mike Nelson, Technical Director

Reviewed by: ______________________ Date: 2/2/98
Monica Carr, Organics Supervisor

Approved by: ______________________ Date: 2/2/98
Karen Kotz, Laboratory Director

Approved by: ______________________ Date: 2/2/98
Harry Romberg, QA Officer

UNCONTROLLED

Table of Contents

1. Introduction and Scope

1.1. Method Description

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC, HPLC, and ion chromatographic determinations. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of normal chromatographic variability.

The width of the retention time windows should be such that the occurrence of both false positives and false negatives is minimized. Retention time windows that are too narrow can result in false negatives or may cause unnecessary reanalysis of sample extracts when surrogate or matrix spike compounds cannot be correctly identified. Conversely, retention time windows that are too wide may result in false positive results that cannot be confirmed by secondary column analysis, or other methods.

This procedure describes the methodology used to establish retention time windows for chromatographic methods. It is based on the practice outlined in SW846, Method 8000. In general terms, standards are analyzed over a time period of no less than 72 hours. Injections made over a period of less than 72 hours may result in retention time windows that are unrealistically small.

The measured retention times of the standards are tabulated and a statistical measure of retention time stability is computed. This measure is then used to set the retention time window half-width used for analyte identification.

In general, a retention time window study is performed during method validation. The retention time windows thus determined are subsequently used for analyte identification during sample analysis. This method is intended to apply to all chromatographic methods performed at Laucks that do not employ a mass spectrometer as the detector: gas chromatography, HPLC, and ion chromatography.

2. Definition of terms and acronyms

2.1. RTW (Retention Time Window)

The width, in minutes, of the retention time window half width. The retention time window for identification is ±RTW.

3. Equipment list and standards

3.1. Equipment

3.1.1. Chromatographic system

The same system that will be used for the analysis of samples or sample extracts. Ensure that the chromatographic system is operating reliably and that conditions have been optimized for the target analytes and surrogate compounds to be determined in the method.

3.2. Standards

Calibration standards required by the method.

4. Safety precautions and waste disposal

4.1. Safety precautions

4.1.1. Standards, samples, and samplesolutions

Handle as if they are hazardous substances.

4.1.2. Instrument operation

Refer to the instrument manufacturer's manual for routine instrument precautions.

Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

4.1.3. Electrical shock

All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

4.2. Waste disposal

4.2.1. All waste disposal precautions and procedures will be detailed in the appropriate analysis SOP.

4.2.2. Waste segregation and disposal from the point of collection is further covered in the appropriate Laucks SOP.

5. Calibration and quality control

5.1. Calibration

Calibrate the instrument as described in the analysis SOP. Before establishing windows, make sure that the chromatographic system is operating under optimal conditions.

6. Operation procedures

6.1. Data collection

For the data collected over the course of at least one analytical sequence no less than 72 hours long, tabulate the analyte retention times for all method target analytes and surrogate compounds from all standards analyzed.

> **Note**
>
> A single analytical sequence is the minimum requirement. In most cases it is advisable to collect data over additional sequences in order to capture a data set of retention times which more closely models real world operating conditions.

To mimic real world operating conditions the standards must bracket sample extracts just as in a normal sample analysis sequence. Collect data for a minimum of 3 standards in each sequence. For multi-response analytes such as Aroclors, select the same peaks which will be used for compound identification.

Record the retention time for each single component target analyte, surrogate spiking compound, and multi-component representative peak to 3 decimal places.

6.2. Calculations

Compute the mean and standard deviation of the measured retention times for each compound using the following equations.

6.2.1. Mean

$$\bar{t} = \frac{\sum_{1}^{n} t_i}{n}$$

6.2.2. Standard deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(t_i - \bar{t}\right)^2}{n-1}}$$

where

t_i=measured retention time

$\bar{t}$ =average retention time

n=number of measurements

6.2.3. Retention time window

$$\mathrm{RTW} = \bar{t} \pm 3 * SD$$

6.2.4. Frequency of RTW determination

This study must be repeated whenever there is a major change to the method such as a new column, a new instrument temperature program, a new gradient program, major instrument overhaul, etc. It is desirable that multiple instruments running the same method have identical RTWs. However, this must be verified experimentally.

6.2.5. Constraints on the experimental determination of RTWs

6.2.5.1. RTW too small

In spite of the effort to mimic real world operating conditions by performing this study using a real analytical sequence, the RTWs may be unrealistically small, even 0.000 minutes, to the limits of the chromatography system. In that case use one of the following methodologies to administratively set the RTWs. When applying one of the administrative methodologies, the analyst's judgment weighs heavily. The desired result is that the RTWs be set such that the window half-width is sufficient to ensure that the chance for both false negatives and false positives is minimized.

6.2.5.1.1. Method 1

Collect additional data and re-compute the RTWs.

Note:

Collecting data over more than one 72-hour analytical sequence should preclude a necessity to use Method 1.

6.2.5.1.2. Method 2

Set the RTW using the following guidelines.

Run Type	RTW half-width, minutes
Narrow bore, megabore capillary	0.03
HPLC	0.15 for analytes with RT to 15 min, 0.20 thereafter (see following notes)
Ion chromatography	0.15 for analytes with RT to 15 min, 0.20 thereafter (see following notes)

Notes:

In the determination of pesticides/PCBs using CLP methodology, the RTWs are fixed in the Statement of Work. This methodology will not apply to pesticides/PCBs determined using the CLP SOW.

The default retention time window half-width (0.03 minutes) that is called out in SW-846 is unrealistically small for HPLC determinations. The numbers listed in the table above are based on Laucks experience with these methods. In most cases, there will be sufficient retention time variation for HPLC analytes that this option will not be necessary.

This method is also implemented for ion chromatography. Ion chromatography is not addressed in SW-846, method 8000.

6.2.5.2. RTW too large

IF, in the analyst's judgment, the compound RTW is too large THEN compute a pooled standard deviation using the following equation. Using this pooled estimate, re-compute the RTW as ±3 times the pooled estimate.

$$S_{pooled} = \sqrt{\frac{\sum_{1}^{k} s_k^2 (n_k - 1)}{\sum_{1}^{k} (n_k - 1)}}$$

k=k*th* set

s_k^2 =variance of k*th* set

n_k =replicates in k*th set*

7. Application to routine analytical sequences

7.1. Setting the RTW for each analytical sequence

The analytical method for each SOP covers this in more detail, but the general methodology is as follows. After the analysis of either the initial multi-point calibration standards or the analysis of the initial calibration verification standard, the RTWs for analyte identification in that analytical sequence are reset using the retention times of the mid-point calibration standard as the center of the window.

7.2. Analyst discretion in analyte identification

Irrespective of the RTW established for analyte identification, the judgment of the analyst weighs heavily in the interpretation of chromatograms. Sample-specific effects can alter the observed retention times of target analytes in sample extracts. In such cases, it is acceptable for the analyst to determine that the target analytes are outside the established RTWs.

Some techniques that are used for such identifications are retention time ratios of target analytes to surrogate compounds, observation of retention times of target analytes in MS/MSD samples, or re-analysis of sample extracts after spiking the extract with target analytes and observing an increase in peak response. Whatever methodology is used must be completely documented in narrative comments for the sample set.

7.3. Corrective actions

The surrogate compounds added to each sample, blank, QC sample, and calibration standards are used to monitor retention time shifts. IF a surrogate compound's retention time falls outside the expected RTW THEN the analyst must determine the cause and correct the problem before proceeding with further determinations.

Note
In some cases sample-specific matrix effects may result in uncorrectable retention time shifts of surrogate compounds. These effects must be documented on a sample by sample basis and the corrective action used for compound identification documented.

The retention times for all target analytes in continuing calibration standards must fall within the established windows. IF retention times drift outside the established windows THEN perform instrument maintenance, analyze a new initial calibration verification standard, and reset the RTW centers.

8. Reports

8.1. Original data

The appropriate analytical department will retain the original files of all RTW studies.

8.2. Working copies

Summarized, tabulated retention time window results will be maintained in the appropriate chromatography laboratory, on an instrument specific basis. On a project specific basis and by request only, these summarized, tabulated RTW results will be provided in the case narrative report.

In addition, these retention time window study summaries will be signed off by the department manager and the summary report scanned and stored in LaserFiche.

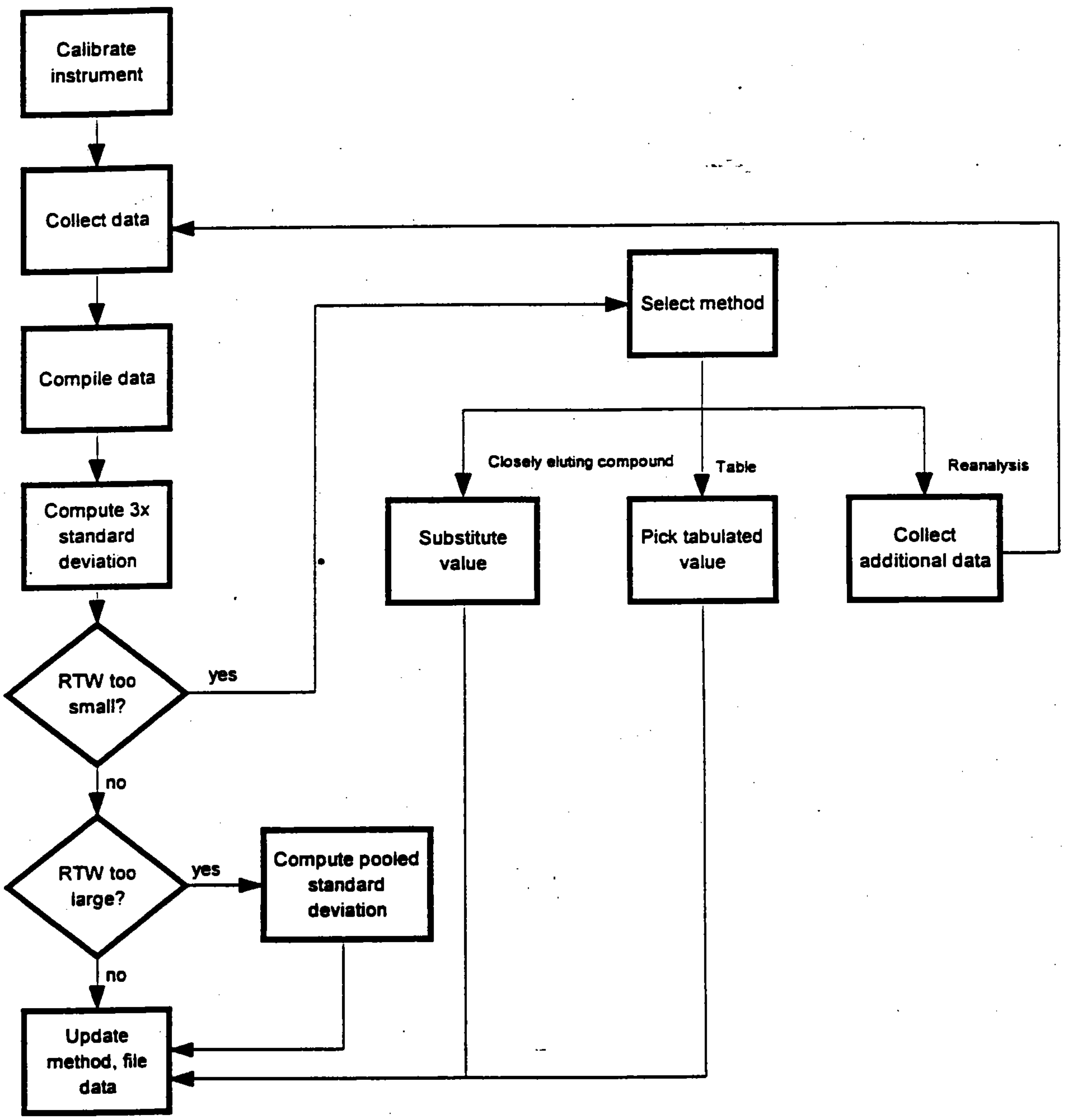
References:

John Keenan Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, 1989

USEPA, *Test Methods for Evaluating Solid Waste*, SW-846, 8000B, various analytical methods.

9. Appendix 1. Flow Chart

Method LTL-8000



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8084

Title: **Analysis of Organochlorine Pesticides and PCBs by SW846 Methods 8081A and 8082**

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Monica Carr, Organic Division Manager

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Harry Romberg, QA Officer

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1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the procedures and specifications for instrumental analysis of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in water and soil following SW846 Methods 8081A and 8082. Analysis is performed by gas chromatography using a single injection port and splitting into dual GC columns with electron-capture detectors. This system provides quantitation and confirmation of pesticides and Aroclors (PCBs) from a single injection. The following table lists the compounds that may be determined by these methods. Additional compounds, listed in Method 8081A, may also be determined.

SW8081A ROUTINE COMPOUNDS	
alpha-BHC	beta-BHC
delta-BHC	gamma-BHC (Lindane)
Heptachlor	Aldrin
Heptachlor epoxide	Endosulfan I
Dieldrin	4,4'-DDE
Endrin	Endosulfan II
4,4'-DDD	Endosulfan sulfate
4,4'-DDT	Methoxychlor
Endrin aldehyde	Endrin ketone
alpha-Chlordane	gamma-chlordane
Toxaphene	
SW8081A OPTIONAL COMPOUNDS	
**Chlordane (not otherwise specified)	Isodrin
Simazine	Atrazine

**When chlordane is requested, a technical chlordane multicomponent standard is analyzed and response factors are calculated for 3-5 major peaks (including the alpha- and gamma-isomers). If these peaks are present in the sample at similar ratios to those found in the technical chlordane standard, an average concentration is calculated from the peaks chosen for calibration. If the peaks are present in the sample at dissimilar ratios to those found in the technical chlordane standard, a calibration factor will be calculated for the standard based upon the sum of the responses for each of the peaks and the sample concentration will be calculated based on the sum of the same peaks in the sample.

SW8082 COMPOUNDS	
Aroclor-1016	Aroclor-1221
Aroclor-1232	Aroclor-1242
Aroclor-1248	Aroclor-1254

Aroclor-1260	

1.1.2 Method Deviations: The analyte list is based on a combination of Method 8081A and 8082 analytes. Additional analytes are only added if requested by the client(s).

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.1.4 In some instances, samples being analyzed for PCBs will require sulfuric acid cleanup. In instances where samples are being analyzed for both pesticides and PCBs, an aliquot is separated prior to the sulfuric acid cleanup step, and is analyzed for pesticides only. The separation of aliquots prior to this cleanup will prevent the potential loss of target pesticide compounds.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C±2°C. Water samples must be extracted within 7 days of sample collection, soil samples within 14 days of sample collection. All extracts must be analyzed with 40 days of sample preparation.

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier	A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation.

Blank spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all HAZWRAP and NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration.
CF	Calibration factor. The ratio of analyte instrument response to nanograms injected. This term is defined in the same way in both the CLP contract and SW 846.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and blank spikes.
IBLK	Instrument blank. This term is borrowed from CLP. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
MDL standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are in the range of 1x to 4x the empirically determined MDLs on an extract/digest basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set.
QC check standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC period	Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.
RT, Retention time	The time (in minutes) at which a target analyte elutes from a chromatography column.
RT window	Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.

Sequence — A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.

SRM — Standard Reference Material - A material containing known quantities of target analytes in a homogeneous matrix which approximates the matrix of the samples being analyzed. It is used to establish that the analytical process is in control.

2. Equipment List and Standards

2.1 Chromatographic System

2.1.1 This analysis requires a gas chromatograph with a programmable oven, heated injection port, dual electron-capture detectors, autosampler and an electronic data-acquisition system.
Equipment list:
Gas chromatograph (Hewlett-Packard 5890 / 5890A)
Autosampler (Hewlett-Packard 7673A)
2 electron-capture detectors (Hewlett-Packard)
2 capillary chromatographic columns of dissimilar phase (J&W DB5 and DB608, 30m x 0.53mm megabore, or equivalents).
Helium carrier gas
5% methane/95% argon detector make-up gas

2.2 Standards

2.2.1 Commercially prepared, certified stock standards are used to prepare working solutions for all surrogates, calibration mixes, and spike mixes. The 2 calibration mixes (INDA and INDB) contain all of the individual pesticide analytes. The spike mix contains 6 representative individual pesticides. These are ordered from Restek, Supelco or an equivalent supplier. Stock standards must be replaced by the manufacturers expiration date.

2.2.2 Calibration standards are prepared at 5 different concentration levels by dilution of the stock standards with hexane. High level INDA and INDB mixtures are made at 16 times the PQL concentration. The lower levels are diluted by factors of 2, 4, 8 and 16. They must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.2.3 Multicomponent analyte calibration standards (Aroclors, chlordane, Toxaphene), with the exception of Aroclors 1016 and 1260, are prepared at one concentration level, by dilution of commercially-purchased stock solutions in hexane. Standards of the Aroclors 1016 and 1260 are combined in a mixture and prepared at five concentration levels. These must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.2.4 A breakdown evaluation (EVAL) mix is prepared from dilution of stocks of 4,4'-DDT and endrin with hexane.

2.2.5 A surrogate mix is prepared from neat materials and contains 2,4,5,6-tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB). These standards are made in hexane and added to all standard mixes (before dilution if applicable).

2.2.6 Refer to SOP LTL-1013 (Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials) for detailed instructions on standards preparation and storage.

2.2.7 Appendix I details the compounds and concentrations contained in all solutions.

3. Safety precautions and Waste Disposal

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they contain hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 The electron-capture detectors used in this analysis contain a radioactive source and should not be opened or otherwise tampered with.

3.2 Waste disposal

3.2.1 Solvent wastes generated in using this procedure (expired standards, old sample extracts, rinsates, etc.) should be emptied into the solvent waste container in the fume hood.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 QA Requirements and Corrective Action

4.1.1 Detailed in the following sections are applicable QA requirements and subsequent corrective actions to be applied to this analysis. A summary of these requirements can be found in Appendix IV.

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on MDLs, located in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.3.2 The precision of spike recoveries must meet or exceed the criteria tabulated in SW 846.

4.4 Retention Time Windows

4.4.1 Retention time window studies are conducted following the Laucks SOP on Establishing RTWs and the procedure detailed in SW 846, Method 8000B, which defines a window for each single and multi- component analyte based on the retention times taken from three standard injections over a 72 hour period.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made, whenever the chromatographic method is significantly modified, or whenever a column is replaced.

4.4.3 The calculated retention time window half widths are typically unrealistic values. Therefore RT windows have been administratively set at the values listed in OLM03.1 for megabore analyses (see table below) and at ±0.03 for capillary analyses. These values are typically wider than 3 times the standard deviation as determined above, but are more realistic.

Analyte	Megabore	Capillary
alpha-BHC	±0.05	±0.03
beta-BHC	±0.05	±0.03
gamma-BHC (Lindane)	±0.05	±0.03
delta-BHC	±0.05	±0.03
Heptachlor	±0.05	±0.03
Aldrin	±0.05	±0.03
alpha-Chlordane	±0.07	±0.03
gamma-Chlordane	±0.07	±0.03
Heptachlor epoxide	±0.07	±0.03
Dieldrin	±0.07	±0.03
Endrin	±0.07	±0.03
Endrin aldehyde	±0.07	±0.03
Endrin ketone	±0.07	±0.03
4,4'-DDD	±0.07	±0.03
4,4'-DDE	±0.07	±0.03
4,4'-DDT	±0.07	±0.03
Endosulfan I	±0.07	±0.03
Endosulfan II	±0.07	±0.03
Endosulfan sulfate	±0.07	±0.03
Methoxychlor	±0.07	±0.03
Tetrachloro-m-xylene	±0.07	±0.03
Decachlorobiphenyl	±0.10	±0.03
Isodrin	±0.07	±0.03
Aroclors	±0.07	±0.03
Toxaphene	±0.07	±0.03
Chlordane	±0.07	±0.03

4.5 Breakdown Evaluation (only required when analyzing for pesticides)

4.5.1 At the beginning of each analysis sequence an evaluation (EVAL) mix must also be analyzed. This is a mid-level standard containing 4,4'-DDT and endrin, and is examined for the breakdown products of these analytes (4,4'-DDD, 4,4'-DDE, endrin aldehyde, endrin ketone) which indicate the need for GC system maintenance. The percent breakdown must be less than 15% for both Endrin and 4,4'-DDT.

4.6 Initial Multi-Point Calibration for Pesticides and PCBs

4.6.1 Analyze single component pesticide standard solutions using at least 5 different concentration levels. The lowest concentration should be at a concentration near, but above, the method reporting limit or PQL. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. This step can be omitted if analyzing for PCBs only.

4.6.2 Analyze multi-component pesticide and PCB standard solutions at one concentration level, with the exception of the Aroclor 1016/1260 mixed standard. This standard should be analyzed at 5 different concentration levels. This step can be omitted if analyzing for pesticides only.

4.7 External Standard Calibration

4.7.1 External standard initial calibration data is evaluated by determining the %RSD of the calibration factors.

4.7.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

4.7.3 The calculated CFs are tabulated and the %RSD calculated. There are no compound-specific criteria. All %RSDs must be within 20% for each analyte or averaged across all analytes in the calibration standard mix.

4.7.4 A set of three to five major peaks is selected for each multicomponent analyte. These should be characteristic of the multicomponent analyte in question. Retention time windows and calibration factors are generated for each of the peaks chosen.

4.7.5 Corrective action

4.7.5.1 If the criteria are not met, the instrument must be re-calibrated.

4.8 Initial Calibration Verification

4.8.1 Concentration and/or CF Criteria

4.8.1.1 At the beginning of an analysis sequence analyze a mid-range calibration standard. The computed calibration factor (CF) or concentration measurement must meet the criteria detailed below.

4.8.1.2 Using the appropriate calculation technique (average CF) compute either CFs or concentration values. For linear calibrations the ICV standard can be verified by calculating either the percent difference or the percent drift.

4.8.1.2.1 The percent difference calculation compares the ICV CFs to the mean CFs from the initial multi-point calibration. The percent difference is calculated as follows:

$$\%D = \frac{Cv - \overline{CF}}{\overline{CF}} \times 100$$

where:

C_v = Calibration Factor

$\overline{CF}$ = Mean Calibration Factor

4.8.1.2.2 The percent drift calculation compares the ICV calculated concentrations to the theoretical (or actual) concentration of the ICV standard. The percent drift is calculated as follows:

$$\%D = \frac{C_c - C_T}{C_T} \times 100$$

where:

C_C= Calculated Concentration
C_T = Theoretical Concentration

4.8.1.3 There are no compound-specific criteria. The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.8.2 Corrective action

4.8.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.9 Updating Retention Time Windows

4.9.1 The retention time windows for compound identification are updated using the retention times for each target analyte in the ICV standard as the center of the window and the previously determined retention time window half-width to establish the retention time range to be used for compound identification.

4.10 Instrument Blank

4.10.1 Criteria

4.10.1.1 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK or solvent rinse. This IBLK analysis is used only to make a judgment as to the possibility of carry-over into the sample extract immediately following the IBLK. Evaluation criteria are detailed below.

4.10.2 Corrective action

4.10.2.1 IBLKs used to monitor for possible carryover in high concentration extracts (those IBLKs optionally placed into the sequence following suspected high concentration extracts) are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be re-analyzed if there is a detectable amount of the analyte found in the IBLK.

4.11 Continuing Calibration Verification

4.11.1 A mid-range calibration standard is analyzed at the frequency of every 10 samples, or every 12 hours, whichever is sooner. In addition, this standard must be the last injection made in the analysis sequence.

4.11.2 Criteria

4.11.2.1 After every 10 sample extract injections, a CCV standard is analyzed. The CF or concentration for each analyte is calculated and the percent difference or percent drift is calculated as shown above.

4.11.2.2 The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.11.2.3 The retention times for all target analytes must fall within the RT windows established by the ICV.

4.11.3 Corrective action

4.11.3.1 Check calculations or perform instrument maintenance. To validate the quantitation of target analytes in analytical samples, the samples must be bracketed by in-control CCVs. However, CCV CFs can be outside the control limits as long as this was due to an increase in response and the corresponding samples contain no detectable levels of the target analyte for which the CF is out of control.

4.12 Method Blanks

4.12.1 Criteria

4.12.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or one blank every 20 samples which ever is more frequent. Any analyte response above the reporting limit is reported.

4.12.2 Corrective action

4.12.2.1 Corrective action may necessitate re-extraction of the sample set. For example, if an analyte were found in the blank but not in any of the associated samples then the sample group may not require re-extraction. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.13 Blank Spikes

4.13.1 Criteria

4.13.1.1 A blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a blank spike. A blank spike is the same as a QC check standard. Blank spike recoveries must meet the criteria specified in the quality control database, QC_DB.

4.13.2 Corrective action

4.13.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re- extraction and re-analysis would be triggered by an out of control blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

4.14 Matrix Spike

4.14.1 Criteria

4.14.1.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{MS - SA}{S} x100$$

where:

MS = concentration in spiked sample
SA = native concentration in unspiked sample
S = spike amount

4.14.1.2 The recovery criteria are detailed in the QC database, QC_DB. In the instance that the native target analyte concentration is greater than 5x the spike concentration, the MS recovery control limits do not apply. In this case, treat the MS/MSD pair as duplicates and report them as such in the quality control database.

4.14.2 Corrective action

4.14.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch-blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.15 Matrix Spike Duplicate

4.15.1 Criteria

4.15.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$\%RPD = \frac{|\chi - \delta|}{(\chi + \delta)/2} x100$$

where:
χ = measured concentration for MS sample
δ = measured concentration for MSD sample

4.15.1.2 RPD control limits are detailed in the QC database, QC_DB.

4.15.2 Corrective action

4.15.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

4.16 Surrogate Recovery

4.16.1 Criteria

4.16.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\% \text{ recovery} = \frac{S_m \times 100}{S_a}$$

where:

S_m = concentration of surrogate measured in extract
S_a = concentration of surrogate added

4.16.1.2 Detailed surrogate recovery control limits are tabulated in the QC database, QC_DB.

4.16.2 Corrective Action

4.16.2.1 Check calculations for possible error. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recovery since non-GC/MS methods cannot separate co-eluting interferences. Hence corrective action is not required for high surrogate recoveries.

4.16.2.2 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and re-analyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

5. Operation procedures

5.1 Chromatographic Conditions

5.1.1 The following general operating parameters are used on gas chromatographs to perform this method utilizing megabore (0.53 or 0.45mm) columns:

Carrier gas:	helium
Column flow:	8 mL/min
Make-up gas:	argon/methane (95%/5% High Purity grade)
Make-up flow:	70 mL/min
Injector:	Grob-type, splitless
Injector temperature:	205°C
Injection:	splitless
Injection volume:	2μL (split - 1 μL per column)
Initial temperature:	150°C
Initial hold time:	0.5 min
Temperature ramp:	4°C per min
Final temperature:	275°C
Final hold time:	9 min
EC detector temperature:	350°C

These GC conditions should be optimized for analyte separation and sensitivity with a particular pair of columns and detectors. Once optimized, the same conditions must be used for the analysis of all standards, samples, blanks and spikes.

5.2 Sample Analysis

5.2.1 Analysis sequence

5.2.1.1 See Appendix II for a detailed analysis injection sequence.

5.2.2 Compound Identification

5.2.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the column 1. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Retention time windows are established as previously described and the absolute retention times are updated each QC period. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes.

5.2.2.3 Identification of multicomponent analytes occurs when the retention times and ratios of each of the multicomponent peaks present in the sample match the peaks chosen for the particular multicomponent analyte in the initial calibration. These same conditions must be met on both columns in order to confirm the presence of the multicomponent analyte. The experienced analyst's judgment weighs heavily in evaluating the patterns of multicomponent analytes with regards to weathering and matrix interferences.

5.2.3 Compound Quantitation

Target compound concentrations are calculated using the following equations:

5.2.3.1 Aqueous samples

5.2.3.1.1 The external standard equation, as expressed in SW 846 is:

$$\text{Concentration}\ (\mu g / L) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(V_s)}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
CF_m = Multi-point average CF
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was diluted 100 µL to 1000 µL and subsequently diluted an additional 100 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t = Volume of total extract, µL.
V_s = Initial sample size, mL.

The reported concentration for multicomponent analytes calculated is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.3.1.2 To report concentrations in alternate units, apply an appropriate factor:

$$mg/L = \mu g/L * 0.001$$

5.2.3.2 Non-aqueous samples

5.2.3.2.1 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Conc.(\mu g / kg) = \frac{(A_s)(V_t)(D)}{(CF_m)(V_i)(W)(TS/100)}$$

where:

W = Weight of sample extracted or purged, grams.

TS = Total solids, percent.

The reported concentration for multicomponent analytes is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.4 Sample Dilution

5.2.4.1 If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. The dilution should be made so that the concentration of the analyte requiring dilution is in the mid to upper calibration range.

6. Reports

6.1 Data Packet Organization

6.1.1 See Appendix III for a check list detailing data packet organization.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the quality control data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and an MS/MSD report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the reporting limit indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
P	When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. If the results from one column is significantly higher (25%) the chromatogram is checked for overlapping peaks, or irregular baseline integration. If no anomalies are discovered, the higher result is reported in order to employ the conservative approach relative to protection of the environment.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
C	The target analyte's presence was confirmed by GC/MS.

6.4 Control Chart(s)

6.4.1.1 The recovery values for gamma-BHC, Heptachlor, Aldrin, Aroclor 1260, TCMX, and DCB in the Blank Spike are plotted on control charts.

7. References

1). U.S. EPA Contract Laboratory Program Contract, Exhibit D, Section III, March 1990

2). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method 8000B, Gas Chromatography, Revision 2, December 1996.

3). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Organochlorine Pesticides by Gas Chromatography, Method 8081A, Revision 1, December 1996 .

4). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Polychlorinated Biphenyls, (PCBs) by Gas Chromatography, Method 8082, Revision 0, December 1996 .

APPENDIX I

Standard Solution Concentrations, µg/L

Compound	STD1	STD2	STD3	STD4	STD5
alpha-BHC	5.0	10.0	20.0	40.0	80.0
beta-BHC	5.0	10.0	20.0	40.0	80.0
delta-BHC	5.0	10.0	20.0	40.0	80.0
gamma-BHC (Lindane)	5.0	10.0	20.0	40.0	80.0
alpha-Chlordane	5.0	10.0	20.0	40.0	80.0
gamma-Chlordane	5.0	10.0	20.0	40.0	80.0
Heptachlor	5.0	10.0	20.0	40.0	80.0
Aldrin	5.0	10.0	20.0	40.0	80.0
Heptachlor epoxide	5.0	10.0	20.0	40.0	80.0
Endosulfan I	5.0	10.0	20.0	40.0	80.0
Dieldrin	10.0	20.0	40.0	80.0	160
4,4'-DDE	10.0	20.0	40.0	80.0	160
Endrin	10.0	20.0	40.0	80.0	160
Endosulfan II	10.0	20.0	40.0	80.0	160
4,4'-DDD	10.0	20.0	40.0	80.0	160
Endosulfan sulfate	10.0	20.0	40.0	80.0	160
4,4'-DDT	10.0	20.0	40.0	80.0	160
Methoxychlor	50.0	100	200	400	800
Endrin aldehyde	10.0	20.0	40.0	80.0	160
Endrin ketone	10.0	20.0	40.0	80.0	160
Isodrin	12.5	25.0	50.0	100	200
Chlordane - technical					1000
Toxaphene			500		
Aroclor-1016	100	250	500	1000	2000
Aroclor-1221			500		
Aroclor-1232			500		
Aroclor-1242			500		
Aroclor-1248			500		
Aroclor-1254			500		
Aroclor-1260	100	250	500	1000	2000
2,4,5,6-tetrachloro-m-xylene	5.0	10.0	20.0	40.0	80.0
Decachlorobiphenyl	10.0	20.0	40.0	80.0	160

Appendix I (Continued)

Calibration Stock Solutions, mg/L

Compound	Mix	Conc
alpha-BHC	A	5.0
beta-BHC	B	5.0
delta-BHC	B	5.0
gamma-BHC (Lindane)	A	5.0
alpha-Chlordane	B	5.0
gamma-Chlordane	B	5.0
Heptachlor	A	5.0
Aldrin	B	5.0
Heptachlor epoxide	B	5.0
Endosulfan I	A	5.0
Dieldrin	A	10.0
4,4'-DDE	B	10.0
Endrin	A	10.0
Endosulfan II	B	10.0
4,4'-DDD	A	10.0
Endosulfan sulfate	B	10.0
4,4'-DDT	A	10.0
Methoxychlor	A	50.0
Endrin aldehyde	B	10.0
Endrin ketone	B	10.0
Isodrin	A	25
Chlordane - technical	TChlor	1000
Toxaphene	Tox	500
Aroclor-1016	AR1660	100
Aroclor-1221	AR1221	100
Aroclor-1232	AR1232	100
Aroclor-1242	AR1242	100
Aroclor-1248	AR1248	100
Aroclor-1254	AR1254	100
Aroclor-1260	AR1660	100

*-The single component pesticides are contained in 2 separate mixes (A and B) when utilizing megabore (0.53 or 0.45mm ID) columns. These same components are combined into 1 mix when utilizing capillary (0.25mm ID) columns.

Breakdown Check Solution (CLP PEM solution), µg/L

Compound	Conc
4,4'-DDT	100
Endrin	50

Surrogate Stock Solution, mg/L

Compound	Conc.
2,4,5,6-tetrachloro-m-xylene	10.0
Decachlorobiphenyl	10.0

APPENDIX II

Analysis Sequence

Injection	Sample
	hexane rinse
1	breakdown check standard (PEM)
2	ICV standard INDAM
3	ICV standard INDBM
4	ICV standard multicomponents(for PCB only analysis - can replace above 3 stds)
5	up to 10 subsequent sample or QC extracts
15	solvent rinse or IBLK (optional - not required)
16	CCV standard INDAM
17	CCV standard INDBM
18	CCV standard multicomponents (for PCB only analysis - can replace above 2 stds)
19	up to 10 subsequent sample or QC extracts
last	CCV standard INDAM
last	CCV standard INDBM
last	CCV standard multicomponents (for PCB only analysis - can replace above 2 stds)

APPENDIX III

Data Packet Order

I. QC SUMMARY

______ Analysts 'Client' Comment (hard copy and floppy)
______ Surrogate Recovery Summary Report
______ Blank Spike Report
______ MS/MSD Report
______ Method Blank Summary

II. SAMPLE DATA:

_____ Organic Analysis Data Sheet
_____ Sample Confirmation Worksheet
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

III. STANDARD DATA:

_____ Linearity Report

Lineanity Standards:
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

Continuing Calibration Standards:
_____ CCV Report
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

_____ Method Blank
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Blank Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike Duplicate
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

V. Bench Sheets

______ Injection Sequence
______ Target Method
______ Extraction Bench Sheets
______ Miscellaneous Work Sheets. i.e. %TS, SDG summary, calculations, HTVR
______ Standards Logs

VI. Reject Data:

DO NOT COPY DO NOT PAGINATE

Data not used to support sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8081A QA Requirements and Corrective Actions:

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	5 calibration stds, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes.	5 calibration stds, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes.	After performing major instrument maintenance or when persistent difficulties meeting CCV criteria occur.	recalibrate or narrate faults if possible	narrative
Initial Calibration Verification	Must be ±15% D for each analyte or averaged across all analytes.	Must be ±15% D for each analyte or averaged across all analytes.	at start of each new analytical sequence, or daily if RTs shift	rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	narrative
Continuing Calibration Verification	Must be ±15% D for each analyte or averaged across all analytes.	Must be ±15% D for each analyte or averaged across all analytes.	Every 10 samples	rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	narrative
Breakdown	Endrin and DDT breakdown must be <=15%	Endrin and DDT breakdown must be <=15%	At the beginning of each sequence.	Rerun, instrument maintenance	narrative
Method Blank	Presence of any target analytes must be below MDL	Must be below reporting limit	every analytical batch or 1 per 20 samples	Report quantitated contaminants with a "B" flag, or re-extract if necessary	case narrative and corrective action form if re-extracted
Surrogate Recovery	Limits to be determined by the lab	listed in QC database	every sample	Re-extraction of the sample is required if any surrogate is out of control. All surrogates must be in control in the method blank, otherwise all associated samples must be re-extracted. Where contractually required, all surrogates must be within control limits.	case narrative and corrective action form if re-extracted
Matrix Spike Recovery	Limits not specified	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted

MS/MSD RPD	Limits not specified	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative corrective action form if re-extracted
Blank Spike Recovery	Limits not specified	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
Standard Reference Material (SRM) Recovery	none required	none required	only if requested	reanalyze or re-extract and reanalyze SRM	case narrative and corrective action form if re-extracted

APPENDIX V

Compound Elution order on DB5 and DB608 Megabore (0.45 or 0.53 mm ID) Columns

DB5	DB608
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
beta-BHC	gamma-BHC (Lindane)
gamma-BHC (Lindane)	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Isodrin	Isodrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
Endosulfan I	alpha-Chlordane
alpha-Chlordane	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
Endosulfan II	4,4'-DDD
4,4'-DDD	Endosulfan II
Endrin aldehyde	4,4'-DDT
Endosulfan sulfate	Endrin aldehyde
4,4'-DDT	Endosulfan sulfate
Endrin ketone	Methoxychlor
Methoxychlor	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

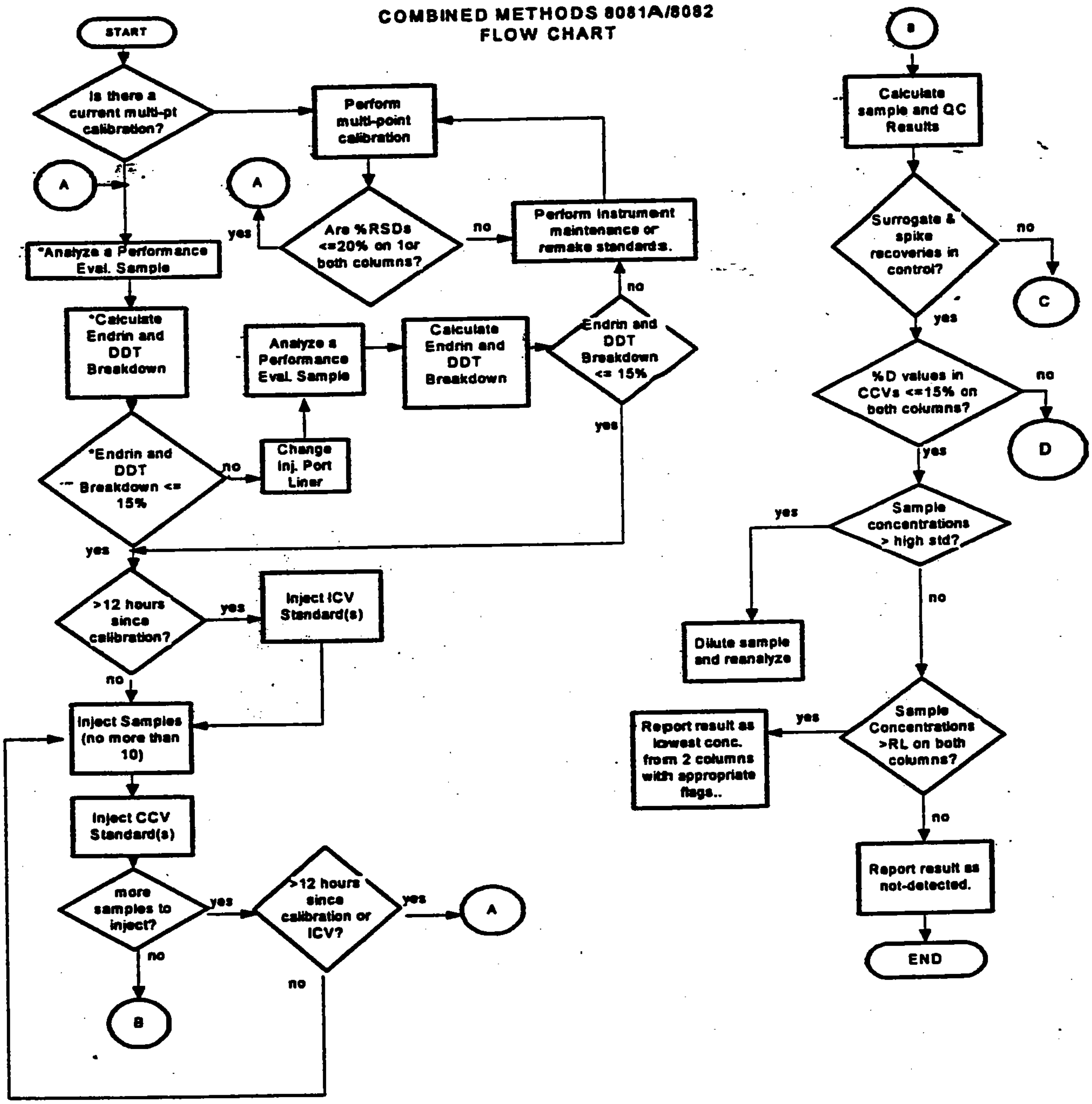
APPENDIX VI

Compound Elution order on DBXLB and DB17ms Capillary (0.25 mm ID) Columns

DBXLB	DB17ms
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
gamma-BHC (Lindane)	gamma-BHC (Lindane)
beta-BHC	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Isodrin	Isodrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
alpha-Chlordane	alpha-Chlordane
Endosulfan I	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
4,4'-DDD	4,4'-DDD
Endosulfan II	Endosulfan II
Endrin aldehyde	4,4'-DDT
4,4'-DDT	Endrin aldehyde
Endosulfan sulfate	Endosulfan sulfate
Methoxychlor	Methoxychlor
Endrin ketone	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

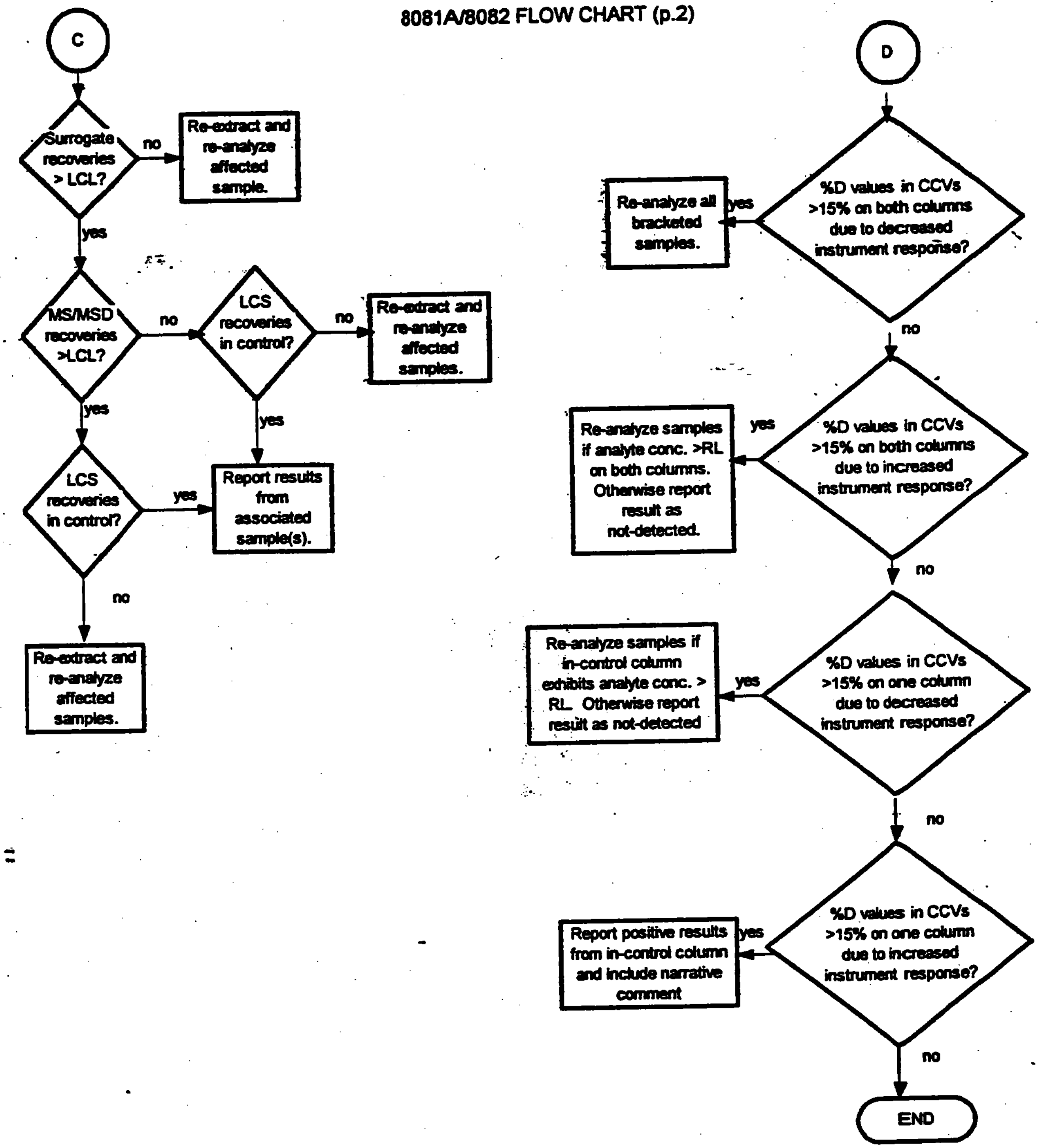
APPENDIX VII

Flow Chart



*These steps can be eliminated if analyzing for SW8082 compounds only

COMBINED METHODS
8081A/8082 FLOW CHART (p.2)



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8277

Determination of Polynuclear Aromatic Hydrocarbon Compounds by Selective Ion Monitoring (SIM) Method 8270C

UNCONTROLLED

Revision history:

Number	Date
0	02/05/98

Written by: Galina Gringer Date: 2/9/98
Galina Gringer, GC/MS Semivolatile Chemist

Approved by: Harry Romberg Date: 2/9/98
Harry Romberg, QA Officer

Approved by: Karen J. Kotz Date: 2/9/98
Karen Kotz, Laboratory Director

TABLE OF CONTENTS

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the determination of polynuclear aromatic hydrocarbon analytes in aqueous, soil, sediment, and other matrices by Selective Ion Monitoring (SIM) method. The SIM analysis scans for only a selected group of ions instead of a whole range of 35 to 500 m/z. The difference in scan time results in increased detector sensitivity, which allows us to report target analytes at very low levels.

1.1.2 This SOP follows SW-846 Method 8270C except for the specific deviations listed below or outlined in a project's specific QAPP.

1.1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas/liquid/chromatography/mass spectroscopy and in the interpretation of chromatograms and mass spectra. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations and comments of Method 8270C, as published, which are followed as standard operating procedure in the performance of this method at Laucks:

- Method 3611B (Alumina Column Cleanup and Separation of Petroleum Wastes) is utilized routinely for all soil/sediment sample extracts. Alumina cleanup is performed on aqueous sample extracts as deemed necessary.

- The 5 initial calibration levels have been established from 0.04ug/mL to 8.0ug/mL in order to demonstrate linearity for all target analytes and to provide a low-level standard that will act as the reporting limit as outlined in the method.

- Neutral surrogate compounds 1-Fluoronaphthalene, Fluorene-d10, and Pyrene-d10 are used routinely. Current surrogate recoveries are maintained in Laucks' quality control database (QC_DB).

- The concentration of internal standards added to all sample extracts and calibration standards has been decreased from 40 ng/uL per compound to 1.0 ng/uL in order to accommodate 2 μL injection volumes as allowed by the method.

- Acceptable retention times used for internal standards in all analyses is +/- 0.50 minutes relative to the daily CCV standard. This range is considerably narrower than the 0.06 RRT units specified in method 8270C and is considered more likely to ensure acceptable method performance.

- Laucks uses a relative response factor for all analytes. The method specifies that if the average RSD of all analytes for the initial calibration is ≤ 15%, then the RRF may be used for individual analytes with RSDs >15%. This method option will be used if any analyte's RSD exceeds 15% in the initial calibration.

- 8270 method DFTPP tuning criteria has been substituted as allowed by the method. Tuning criteria is listed in Appendix II.

- All standards are stored at -10°C or by the manufacturer's recommendation. Sample extracts are stored at 4°C.

- The surrogate and matrix spikes will be added to the sample such that the final amount injected from normally concentrated samples is 5 ng for all spiking analytes.

- For several ongoing projects Laucks uses relative response factors from the continuing calibration verification to quantitate the concentration in the sample. For future projects Laucks will use an average response factor from the initial calibration to calculate concentration from the sample.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C. Water samples must be extracted within 7 days of collection. Soil samples must be extracted within 14 days of collection. All sample extracts must be analyzed within 40 days of sample preparation.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all HAZWRAP and NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard.
CCC	Calibration Check Compound. A compound of analytical interest whose RRF in the CCV is compared to the average RRF from the initial calibration. The % difference must be less than the value specified in the method for the CCV to be considered valid. CCCs must also meet maximum %RSD criteria in an initial calibration.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IPCS	Instrument Performance Check Solution. A solution containing at a minimum DFTPP, pentachlorophenol, benzidine, and p,p'-DDT. The IPCS is analyzed at the start of a 12 hour QC period in order to verify DFTPP tuning criteria.
Internal Standard	A compound added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a know concentration, prior to analysis. Internal standards are used as the basis for quantitation of target compounds.

MDL — Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.

MDL Standard — Method detection limit standard. A standard prepared so that the concentrations of the target analytes are approximately 4x the empirically determined MDLs on an extract basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.

QC Period — Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts. A QC period is 12 hours starting with the injection of the CCV standard or DFTPP performance evaluation.

RF — Response Factor. The measure of the mass spectral response of an analyte compared to its internal standard. Response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RF is determined by the following equation:

$$RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:

A_x - Area of target analyte primary ion
C_{is} - Concentration of internal standard
A_{is} - Area of internal standard primary ion
C_x - Concentration of target analyte

RSD or %RSD — Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

RT — Retention time. The time (in minutes) at which a target analyte elutes from the GC column.

Sequence — A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

SIM — Selective Ion Monitoring. This is a type of analysis when the MS detector is programmed to scan for only the selected ions.

Surrogate — Compounds added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a known concentration; used to evaluate extraction and analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental samples.

2. Equipment List and Standards

2.1 Chromatographic System

Gas Chromatograph: Hewlett Packard 5890 I or II.

Carrier Gas: Helium 99.995% (high purity) or better.

Column: 30 m x 0.25 mm x 0.25 µf capillary column (Restek RTX-5 or equivalent).

Automatic Sampler: Hewlett Packard 7672A with 19405A and 3396A controllers.

GC/MS Interface: Capillary direct to the ion source of the mass spectrometer, fixed temperature.

Mass Spectrometer: Hewlett Packard 5970B.

Data System: Teknivent.

Miscellaneous: Assorted syringes, vials, caps, septa, injection port liners, ferrules, etc.

Note: All of the above equipment may be substituted with equivalent or better equipment.

2.2 Standards

2.2.1 Preparation of Semivolatile Standards

2.2.1.1 All standards prepared from this operating procedure must be logged into one of two standards preparation logbooks. One is maintained for stock solutions prepared from neat chemicals; the other is maintained for all working solutions. These logbooks are kept in the GC/MS semivolatile working area. When a standard is made, a solution number is assigned to it. This solution number is unique and will be used to track and identify the standard every time it is analyzed.

2.2.1.2 An example of the solution nomenclature used is a working PNA SIM standard prepared on 2/20/97. The solution number assigned was MS 5-70-04. This label represents the following:

MS	Solution was made and used as a Mass Spec standard.
5	Solution was logged into standard book #5.
70	Page number on which solution has been recorded.
4	This denotes the fourth entry on page 70.

2.2.1.3 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by purchasing solutions which have been fully documented by a commercial vendor.

2.2.2 Preparation of Internal Standard Solution (PNA SIM IS MIX @ 200 ug/mL).

Internal standards
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12
1,4-Dichlorobenzene-d4 [(A)]
(A) - this compound is present in the mix, but not appropriate to use for PNA analysis, and ,therefore, not reported)

2.2.2.1 Commercially prepared and certified internal standard solution is puchased at a concentration of 2000 µg/ml and is used as diluted 1:10 in methylene chloride. This standard is monitored for degradation by evaporative losses. The standard should be replaced when the area counts increase more than 15% from when it was freshly opened. This standard is kept at -10°C until put into use. Once opened, the standard is kept at room temperature to avoid having the heavier compounds from falling out of solution.

2.2.3 Preparation of Surrogate Standards

Surrogate compounds
1-Fluoronaphthalene
Fluorene-d10
Pyrene-d10

2.2.3.1 Surrogate stock solutions are prepared by dissolving 100 mg of each analyte in 10 mL of methanol resulting in a stock solution with a concentration of 10,000 µg/mL.

2.2.3.2 0.5 mL of each of the PNA surrogate stock solutions are mixed and diluted in methanol to a final volume of 25 mL to make an intermediate working solution of 200 µg/mL. A 1.25 mL aliquot of the intermediate working solution is diluted in 25 mL of methanol to make a working solution of 5.0 ug/mL.

2.2.3.3 An aliquot of working solution is diluted 1:5 in methylene chloride and analyzed by the GC/MS department. The working solution must be within 80% - 120% of the expected values of 1 ng/µL for all three surrogates before they are put into use by the extractions department.

2.2.3.4 Commercially prepared and certified surrogate solution may be purchased and used in place of the above described solutions at the discretion of the laboratory.

2.2.4 Preparation of Matrix Spike Standards

Matrix Spike Compounds

Acenaphthylene	Chrysene
Acenaphthene	Benzo(b)fluoranthene
Fluorene	Benzo(k)fluoranthene
Phenanthrene	Indeno(1,2,3-cd)pyrene
Anthracene	Dibenzo(a,h)anthracene
Fluoranthene	Benzo(g,h,i)perylene
Pyrene	Benzo(a)anthracene
Benzo(a)pyrene	2-Methylnaphthalene

2.2.4.1 Commercially prepared and certified spiking solution SV Mix #5 is purchased from Restek. This standard solution containes all PNAs listed above at 2000 ug/mL in methanol with the exception of 2-methylnaphthalene. 2-Methylnaphthalene stock solution is prepared and verified in the same manner as surrogate standards.

2.2.4.2 625 uL of SV mix # 5 and 125 uL of 2-methylnaphthalene stock are mixed and diluted in 25 mL of methanol to prepare an intermediate working solution of 50 ug/mL. 2.5 mL of an intermediate working solution is diluted to a final volume of 25 mL in methanol to make a working standard solution of 5 ug/mL.

2.2.4.3 An aliquot of the working solution is diluted 1:5 in methylene chloride and analyzed by the GC/MS department. The working solution must be within 80% - 120% of the expected values of 1 ng/µL for all spiking analytes before they are put into use by the extractions department.

2.2.5 Preparation of DFTPP Solution

2.2.5.1 Prepare a solution of DFTPP (Decafluorotriphenylphosphine) at a concentration of 5000 µg/mL in acetone. Store this solution in amber screw-cap vials in the freezer. The vial in use may be stored at room temperature.

2.2.5.2 Commercially prepared and certified DFTPP solution may be purchased and used in place of the above described solution. This solution and working standards made from it are kept at -10°C.

2.2.6 Preparation of Calibration Standards

2.2.6.1 Calibration standards are prepared in methylene chloride from stock solutions which are purchased from a commercial source (e.g., Supelco or Restek). If an analyte required for calibration is not present in an available mixed solution, laboratory-prepared stock solutions which have been verified by GC/MS may be used. Laboratory prepared stock solutions should be tested against independent reference standards when they are available.

2.2.6.2 Calibration standards are prepared at six concentration levels (0.04, 0.4, 1.0, 4.0, and 8.0 ng/µL). Each calibration standard contains all compounds of interest, surrogates, and internal standards. The internal standards are added so that they are present in all calibration standards at a concentration of 2.0 ng/µL each.

2.2.7 Preparation of PNA200 stock solution

2.2.7.1 Assemble the following solutions in order to prepare a combined stock solution which will contain all analytes of interest at a concentration of 200 µg/mL each:

SV Calibration Mix 5 Restek #31011 (contains the following analytes at 2000 ug/mL)

Acenaphthylene	Chrysene
Acenaphthene	Benzo(b)fluoranthene
Fluorene	Benzo(k)fluoranthene
Phenanthrene	Indeno(1,2,3-cd)pyrene
Anthracene	Dibenzo(a,h)anthracene
Fluoranthene	Benzo(g,h,i)perylene
Pyrene	Benzo(a)anthracene
Benzo(a)pyrene	

2-Methylnaphthalene stock (10700 µg/mL) made from a neat (Chem Service)
Carbazol stock purchased from Supelco
Surrogate stock (made of individual standards. Each one was made from a neat.)

2.2.7.2 The vendor and catalog numbers provided are for reference only. Other vendor's certified solutions may be substituted.

2.2.7.3 Combine appropriate amounts of all solutions in a clean, silanized volumetric flask (2.0 or 5.0 mL capacity) so that all analytes are present at 200 µg/mL. Dilute to volume with methylene chloride, stopper the flask and mix well. Record all information in the working standards logbook and transfer the contents of the volumetric flask into silanized amber screw-cap vials. Store this solution in the freezer when not in use. The vial must be marked with the logbook name, standard type, preparation date, solvent used, and expiration date.

2.2.8 Preparation of working calibration standards

2.2.8.1 To prepare working calibration standards, add the amounts listed below (in µL) of PNA200 stock solution or PNA1.0 working solution, internal standard solution (PNA IS MIX @ 200 ug/mL) and methylene chloride to clean vial inserts and use within one week.

Preparation of Working Standards

	Amount Added Standard PNA200 (μl)	Amount IS Added (μl)	Amount CH_2Cl_2 Added (μl)
Working PNA 8.0	8	2	190
Working PNA 4.0	4	2	194
Working PNA 1.0	1	2	197

	Amount Added Standard PNA1.0 (μl)	Amount IS Added (μl)	Amount CH_2Cl_2 Added (μl)
Working PNA 0.4	80	2	118
Working PNA 0.04	8	2	190

2.2.8.2 Log into the working solutions logbook all of the above information as the standards are made. Store all standards at -10°C for up to one week when not in use.

2.2.8.3 The working PNA1 standard is made in larger quantity because it is used every day as a calibration check. It will be necessary to prepare a fresh PNA1 on a weekly basis.

2.2.8.4 Calibration stock solutions which are received sealed in ampules from the manufacturer are useable up to their manufacturer's expiration date. The mixed PNA200 stock solution is stable for up to 6 months or 1 year when promptly ampuled. Working calibration standards may be used for 1 week.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 All waste solvents, expired standards and old extracts should be disposed of in the solvent waste can located in the prep area fume hood. Consult the laboratory SOP for more detail on waste disposal.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Chromatographic Conditions

Initial Temperature:	80°C.
Initial Time:	2 minutes.
Column Temperature Program:	9°C/minute.
Final Temperature:	305°C.
Final Time:	8 minutes.
Injector Temperature:	250°C.
Transfer Line Temperature:	280°C.
Injector Purge Time Off:	42 seconds (0.7 minutes).
Injection Volume:	2 µL.
Column Linear Velocity:	30-40 cm/sec (nominal 35 cm/sec measured at 30°C).

4.1.2 Mass Spectrometer Conditions

Electron Energy:	70 volts.
Scan Time:	Not to exceed 1 second per scan.
Scan Start Time:	4.5 minutes.
Scan Time Range:	from 4.5 to 16.5 minutes.
Scans in Mass Segment:	125 to 177 amu.
Scan Time Range:	from 16.5 to 27 minutes.
Scans in Mass Segment:	165 to 245 amu.
Scan Time Range:	from 27 to 36 minutes.
Scans in Mass Segment:	248 to 280 amu.

4.2 Method Detection Limit Study

4.2.1 MDL studies will continue to be performed. This procedure is fully described in Laucks SOP on Determination of MDL's.

4.3 Method Reporting Limits

4.3.1 The method reporting limit for this method shall be set as described by method 8000B by using the lowest calibration standard as the method report limit. Values detected below this level will be reported but will be "J" flagged as outlined in Section 5.2 of this SOP.

4.4 Method Validation

4.4.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher. This procedure is fully described in Laucks SOP on Determination of Precision and Accuracy Studies.

4.5 Daily Instrument Maintenance

4.5.1 Daily instrument maintenance is required for good chromatography and proper calibration. The following steps must be undertaken as needed before the analysis of any standards or samples.

- Cool GC oven to 30°C.
- Check background.
- Remove injector septum and liner.
- Remove column from injector.
- Install a clean quartz liner.
- Reinstall O-ring or replace if worn.
- Install a new ferrule on the column.
- Clip off 8-10 cm of the column. Check for proper cut.
- Reinstall column in injector and adjust height.
- Install injector cap and a new septum.
- Clean and re-install the autosampler syringe.
- Check the background again.
- If background is okay, ramp the GC oven twice from 30°C to 300°C at 15°C/minute and hold 8 minutes.

4.6 Instrument Tuning

4.6.1 The HP 5970B mass spectrometer uses FC-43 (PFTBA) as a mass calibration compound. Each instrument will require different ion ratios to pass the required DFTPP performance criteria. See Appendix II for the DFTPP tuning criteria. The following ratios are therefore approximate. Use manual tuning to tune the mass spectrometer.

Ion	% of ion 69
69	100%
131	25-35%
219	25-35%
414	1-3%

4.6.2 Ion peak widths should be in the 0.5 to 0.6 amu range. Excessive peak width can lead to loss of minor isotope peaks. Insufficient peak width can result in the loss of sensitivity. Ions for water, nitrogen, and oxygen should be <5% of ion 69. A copy of the PFTBA spectrum and tabular listing should be kept with the instrument historical file.

4.7 Initial Multi-Point Calibration

4.7.1 An instrument performance check solution containing DFTPP should be injected first in order to verify DFTPP tuning criteria, degradation, and column tailing factors. The spectrum of DFTPP must pass tuning criteria before any other standards are injected. Retuning of the instrument may be necessary to achieve this. Additionally, the DFTPP spectrum should be well within the performance criteria - i.e., no ion abundance should be borderline and the ratios should be routinely reproducible to insure a stable calibration.

4.7.2 Analyze standard solutions using five different concentration levels. The lowest concentration should reflect the current report limit being used. The highest concentration should define the upper usable working range of the detector. Analyze the calibration standards following an acceptable DFTPP injection. The initial calibration must meet the criteria outlined later in this SOP.

4.8 Continuing Calibration Verification

4.8.1 At the beginning of a QC period, analyze an instrument performance check solution. The DFTPP tuning criteria in Appendix II must be met prior to analyzing a CCV standard. If DFTPP tuning criteria are met, analyze a CCV standard. The standard analysis must meet the %D and minimum RRF criteria detailed later in this SOP.

4.9 Sample Analysis

4.9.1 Analysis sequence

4.9.2 In general, the method blank accompanying the samples is injected prior to the analysis of the samples. This is not a requirement, however. Samples are then injected until the end of the 12 hour clock. After 12 hours have expired, instrument maintenance is performed and another IPCS and CCV standard are analyzed and evaluated before sample analysis continues.

4.9.3 Extract Preparation

4.9.4 Remove sample extracts from the refrigerator and allow them to come to room temperature.

4.9.5 Transfer 200 μL of extract to a vial insert.

4.9.6 Add 2 μL of IS solution (PNA IS MIX @ 200 ug/mL).

4.9.7 Cap the vial and mix well.

4.9.8 Place the vial onto the autosampler for analysis.

4.9.9 For dilutions, decide what dilution is necessary from previous data or analyst judgment. Make dilution and then add 2 μL of IS solution prior to capping vial. Mix well prior to analysis.

4.9.10 Compound Identification

4.9.10.1 Compounds are tentatively identified if a peak elutes within 0.5 minute of that compound in the CCV standard. In addition, the internal standard for that compound should also be within 0.5 minute of its counterpart in the CCV standard. To confirm the presence of that compound in the sample extract, the mass spectrum of the peak must be evaluated. Spectra are compared against standard spectra of each compound generated on the instrument used for analysis.

4.9.10.2 The following criteria are used to evaluate mass spectra:

- The intensities of the characteristic ions of a compound maximize within one scan of each other. Searches performed based on the presence of a target compound at a compound-specific retention time will be accepted as meeting this criterion.

- All ions present in the standard mass spectra at a relative intensity greater than 30% (most abundant ion in the spectrum equals 100%) or the three ions of greatest intensity must be present in the sample spectrum.

- The relative intensities of ions specified must agree within 30% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.

- If a compound cannot be verified by all of these criteria, but in the technical judgment of the analyst, the identification is correct, then report that identification and proceed with quantification.

- The experienced analyst's judgment weighs heavily in evaluating chromatograms and mass spectra for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. If this has occurred, it must be fully documented in the appropriate report notes.

4.9.11 Common Analytical Problems

4.9.11.1 An analyst's professional judgment plays a large role in how data is interpreted. The following guidelines should be followed in order to facilitate consistency between analysts. Any anomalies not addressed in this SOP must be discussed with the supervisor prior to implementation. All anomalies and corrective actions must be documented.

4.9.12 Carryover

4.9.12.1 In some cases, if analytes in a sample are very high, there may be carryover from one analysis to the next. If a sample is suspected of being high due to historical data or extract color, the sample should be diluted prior to analysis, or one or more blanks should be analyzed after the sample to insure that there is no carryover between analyses.

4.9.12.2 However, in the case where high levels were not expected, and do appear in a sample analysis, the analysis after it should be examined carefully for carryover.

4.9.13 Manual Peak Integrations

4.9.13.1 Manual peak integrations should be used only when necessary to correct for matrix interference, tangent peaks, and rising baselines. Manual integrations are not to be used in an attempt to meet calibration, surrogate recovery, or spike recovery criteria.

4.9.13.2 If the chromatogram shows degradation due to sample loading e.g. split peaks, lift off, or severe tailing, the sample should be diluted and reanalyzed if required detection limits permit.

4.9.13.3 If a manual integration is necessary, follow the following guidelines:

- Integrate only the peak. Start where the peak lifts from the baseline and end as soon as it returns to the baseline. Do not integrate extra baseline in an attempt to increase peak area.

- Do not "peak shave". Do not cut off legitimate parts of the peak in order to reduce peak area.

- In cases of tangent skims, do not increase or decrease peak areas or heights by skimming extra long baselines or drawing the baseline too low.

- Always initial and date your manual integrations.

4.9.14 Compound Quantification

4.9.14.1 Aqueous samples

4.9.14.1.1 The equation for internal standard calculations is

$$Extract\ Concentration(ng/\mu L) = \frac{A_x \times C_{is}}{A_{is} \times RRF}$$

where:

A_x = Response for the target analyte
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard, in ng/µL
RRF = Relative Response Factor (calculated from the CCV)

4.9.14.1.2 The above equation is used directly by the HP computer to yield the extract concentration. To calculate the actual sample concentration, the following calculation must be used.

$$Sample\ Concentration(\mu g/L) = \frac{F \times D \times V_f \times GPC}{V_i}$$

where:

F = Amount found from HP quantitation report (ng/µL)
D = Dilution factor of extract
V_f = Final extract volume (µL)
GPC = GPC dilution factor. Use 1 if GPC was not performed, 2 if GPC was used
V_i = Initial sample volume (mL)

4.9.14.1.3 Normally, these calculations are automatically performed by the LIM system.

4.9.14.1.4 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

4.9.14.2 Non-aqueous samples

4.9.14.2.1 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Sample\ Concentration(\mu g / L) = \frac{F \times D \times V_f \times GPC}{W_s \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

4.9.14.2.2 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

5. Reports

5.1 Data Packet Organization

5.1.1 See Appendix III for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Sample Result Reports

5.3.1 Data Qualifying Flags

5.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U: The analyte of interest was not detected, to the limit of detection indicated.

B: The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.

J: The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.

D: The value reported is derived from the analysis of a diluted sample or sample extract.

X: Indicates an unresolvable isomeric pair. This flag indicates that calculated results are the sum of the two isomers.

E: The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.

6. Quality Control

6.1 General Issues

6.1.1 See Appendix VII and VIII for QA/QC limit and corrective action tables.

6.2 Initial Calibration

6.2.1 Criteria

6.2.1.1 Initial calibration data are evaluated using %RSD of the relative response factors as well as minimum average RRFs. The %RSD must be < 30% for each individual Calibration Check Compound (CCC). All other RRFs must be ≤ 15%. Alternatively, RRFs may be used as long as the average RRFs for all compounds in the method are ≤ 15% and the CCCs are < 30%.

6.2.1.2 Calculate the %RSD for all compounds. CCC criteria are listed in Appendix IV.

6.2.1.3 Calculate the individual and average RFs for each compound.

6.2.1.4 RFs are calculated using the equation

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Response of target analyte
A_{is} = Response of internal standard
C_s = Amount of target analyte (ng/µL)
C_{is} = Amount of internal standard (ng/µL)

6.2.1.5 The minimum acceptable average RF for all analytes is 0.050.

6.2.2 Corrective action

6.2.2.1 If the criteria are not met, other standards may be analyzed or appropriate instrument maintenance and analysis of new standards must be performed. Failure to achieve the required minimum average RRF for the SPCC compounds may indicate the need to perform instrument maintenance or prepare fresh calibration standards.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 At the beginning of each 12 hour QC period, a CCV standard is analyzed. The RF for each compound is calculated and the percent difference is calculated as follows:

$$\%Difference = \frac{RF_c - RF_i}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration.
RF_c = RRF from CCV standard.

6.3.1.2 The %D results for all CCCs must be less than 20%. The %D for any other analytes should be < 30%.

6.3.1.3 In addition, all compounds must have a minimum RRF of at least 0.050. See Appendix V for all CCV criteria.

6.3.2 Corrective action

6.3.2.1 Check that all peaks have been properly integrated. Check the calculations. If the %D criteria are still not met, perform corrective instrument maintenance or re-tuning, and reanalyze the standard. If the %D or minimum RF criteria are still not met, a new CCV standard may need to be prepared, a new initial calibration performed, a new column installed, or other instrument maintenance performed in order to meet the CCV criteria.

6.4 Method Blank

6.4.1 Criteria

6.4.1.1 A method blank is used to verify contamination-free reagents and apparatus. A method blank is prepared with every set of samples extracted at the same time, at a frequency of at least one blank per 20 samples.

6.4.1.2 The concentration in a method blank of any analyte of concern should not be higher than the reporting limit. Any analyte response above the MDL is reported. Values below the reporting limit are "J" flagged. Method blank control limits are detailed in Appendix VI.

6.4.1.3 Where contractually required, e.g. Navy work, all analyte concentrations in the method blank must be <MDL.

6.4.2 Corrective action

6.4.2.1 The blank should be reanalyzed first if carry-over from a previously analyzed sample is believed to be the cause of the contamination. If the contamination is not present in the second analysis, the results of the second analysis may be used. Any similarly affected samples should also be reanalyzed.

6.4.2.2 If an analyte is found in the blank but not in any of the associated samples, the sample batch may not require re-extraction unless this is required by contractual obligations. Consult the QC officer to determine if re-extraction and reanalysis are required.

6.4.2.3 If any analyte exceeds the control limit in the blank and is also found in the associated sample extracts, the samples must be re-extracted and reanalyzed.

6.4.2.4 In any event, it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

6.5 QC Check Sample (LCS)

6.5.1 Criteria

6.5.1.1 The LCS is used to determine whether a method is in control during sample preparation and analysis. A LCS follows the same protocol as the matrix spike analysis except that the spiking solution is added to a blank matrix (deionized water for water or sea sand for soil/sediments) instead of an actual sample. Control limits are maintained in the Laucks quality control database (QC_DB)

6.5.2 Corrective action

6.5.2.1 Check all peak integrations and sample calculations.

6.5.2.2 If the analyte recoveries still exceed the control limits, the blank spike extract should be reanalyzed.

6.5.2.3 If the analyte recoveries are still out of control, re-extraction of the associated samples is required.

6.5.3 Matrix Spike

6.5.3.1 Criteria

6.5.3.2 A sample is chosen from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is not required that a matrix spike analysis be performed with each extraction batch unless the project QAPP requires it. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of selected target analytes. Analyte recovery is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.5.3.3 The control limits for MS/MSD recoveries are available in the Laucks' quality control database (QC_DB).

6.5.4 Corrective action

6.5.4.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases, a narrative explanation of the condition is required to detail the corrective actions taken.

6.6 Matrix Spike Duplicate

6.6.1 Criteria

6.6.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values.

6.6.1.2 This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2)/2} \times 100$$

where:

S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.6.1.3 The control limits for MS/MSD recoveries and RPDs are maintained in the Laucks quality control database (QC_DB).

6.6.2 Corrective action

6.6.2.1 Corrective action for RPD values which exceed the control limits follows the corrective action for MS/MSD recoveries. If more than one RPD exceeds the control limit, re-extraction may not be required if it can be demonstrated that the sample is non-homogeneous and all MS/MSD recoveries are within the control limits.

6.7 Surrogate Recovery

6.7.1 Criteria

6.7.1.1 Surrogates are chemically similar compounds added to every sample, method blank and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follow:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:

S_m = Concentration of surrogate measured in extract.
S_a = Concentration of surrogate added.

6.7.1.2 The control limits for surrogate recoveries recoveries are maintained in the Laucks quality control database (QC_DB).

6.7.2 Corrective Action

6.7.2.1 Check calculations for possible error.

6.7.2.2 Check instrument performance, if necessary correct the problem and re-analyze the extract.

6.7.2.3 Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant matrix interference. This may result in the dilution of the surrogate response to the point that the recoveries can not be measured. If the surrogate recoveries are available from a less-diluted or iundiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. For all package work both the diluted and undiluted analyses will be provided.

6.7.2.4 Re-extraction is not necessary in the case where a sample is chosen for MS/MSD analyses and the same recovery pattern is present in all three analyses. In this case, matrix effects may be assumed and re-extraction is not required for this sample.

6.7.2.5 All other circumstances require re-extraction and reanalysis of the affected sample(s).

6.7.2.6 Out of control surrogate recoveries in the method blank require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique," Revision 2, December 1996, U.S. EPA.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, 2nd Update, Method 3640A, "Gel-Permeation Cleanup," Revision 1, November 1992, US EPA.

US EPA. Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, Document Number OLM03.1, August 1994.

APPENDIX I

Calibration Standard Solution Concentrations, ng/µL

Compound	STD1	STD2	STD3	STD4	STD5
1-Fluoronaphthalene	0.04	0.4	1.0	4.0	8.0
Naphthalene	0.04	0.4	1.0	4.0	8.0
2-Methylnaphthalene	0.04	0.4	1.0	4.0	8.0
Acenaphthylene	0.04	0.4	1.0	4.0	8.0
Acenaphthene	0.04	0.4	1.0	4.0	8.0
Fluorene-d10	0.04	0.4	1.0	4.0	8.0
Fluorene	0.04	0.4	1.0	4.0	8.0
Phenanthrene	0.04	0.4	1.0	4.0	8.0
Anthracene	0.04	0.4	1.0	4.0	8.0
Carbazole	0.04	0.4	1.0	4.0	8.0
Fluoranthene	0.04	0.4	1.0	4.0	8.0
Pyrene-d10	0.04	0.4	1.0	4.0	8.0
Pyrene	0.04	0.4	1.0	4.0	8.0
Benzo(a)anthracene	0.04	0.4	1.0	4.0	8.0
Chrysene	0.04	0.4	1.0	4.0	8.0
Benzo(b)fluoranthene	0.04	0.4	1.0	4.0	8.0
Benzo(k)fluoranthene	0.04	0.4	1.0	4.0	8.0
Benzo(a)pyrene	0.04	0.4	1.0	4.0	8.0
Ideno(1,2,3-cd)pyrene	0.04	0.4	1.0	4.0	8.0
Dibenz(a,h)anthracene	0.04	0.4	1.0	4.0	8.0
Benzo(g,h,i)perylene	0.04	0.4	1.0	4.0	8.0

APPENDIX II

DFTPP Tuning Criteria

Mass	Relative Abundance
51	30-60% of mass 198
68	less than 2% of 69
69	present
70	less than 2% of 69
127	40-60% of mass 198
197	less than 1% of 198
198	100%
199	5-9% of mass 198
275	10-30% of mass 198
365	greater than 1.0% of mass 198
441	0-100 % of mass 443
442	40-100% of mass 198
443	17-23% of mass 442

8270 Method Criteria has been used as allowed by the method. The spectrum must be taken by averaging the peak apex with each of the adjacent scans and background subtracting not greater than 20 scans prior to the beginning of DFTPP.

APPENDIX III

Data Packet Check List

QC Summary
Surrogate Recovery Summary Report
Blank Spike Recovery Report
MS/MSD Recovery Report
Method Blank Summary
GC/MS Instrument Performance Check
Internal Standard Area and RT Summary Report

Sample Data
Target Compound Results (Organics Analysis Data Sheet)
Tentatively Identified Compound (TIC) Results (if required)
Sample Chromatograms, quantitation reports and spectra for all samples

Standards Data
Initial Calibration Summary Report
Chromatograms and quantitation reports for all initial calibration standards
Continuing Calibration Summary Reports
Chromatograms and quantitation reports for all CCV Standards

Raw QC Data
Bar Graph Spectrum, mass listing and chromatogram for every DFTPP injection
Method Blank Data
Target Compound Results (Organics Analysis Data Sheet)
Tentatively Identified Compound (TIC) Results (if required)
Chromatograms, quantitation reports and spectra for all method blanks
Blank Spike Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all blank spikes
Matrix Spike/Matrix Spike Duplicate Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all MS analyses
Control Charts

Bench Sheets
All extraction bench sheets, analysis log book pages, chains of custody, and any other pertinent information

APPENDIX IV

Initial Calibration Criteria

Calibration Check Compound	%RSD Limit
Acenaphthene	< 30%
Fluoranthene	< 30%
Benzo(a)pyrene	< 30%

RSD for all other analytes must be < 15%.

APPENDIX V

Continuing Calibration Verification Criteria

Calibration Check Compound	%D Limit
Acenaphthene	20%
Fluoranthene	20%
Benzo(a)pyrene	20%

%D for all other analytes must be within 30%.

APPENDIX VI

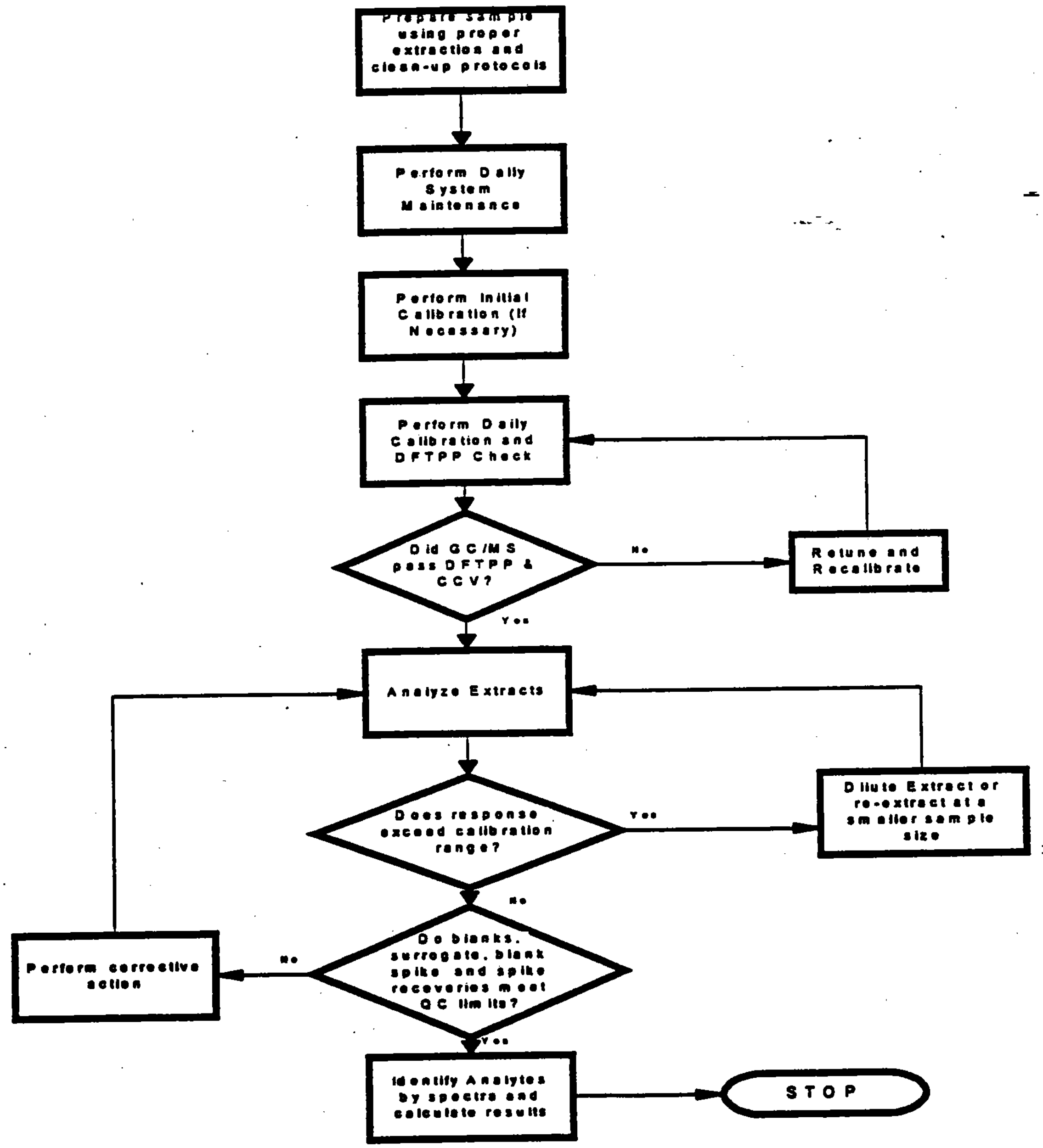
Method Blank Control Limits

	Control Limits	
Compound	Water	Soil
All compounds	<RL	<RL

If contractually required, e.g. Navy work, all analyte concentrations must be < MDL in the method blank.

APPENDIX VII

Method 8270 Flow Chart



APPENDIX VIII

Method 8270C QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Minimum of 5 levels, lowest near but above MDL, %RSD for CCC <30, All others should be <15%.	5 levels, %RSD for CCC <30, All others <15%, All RFs >0.05 Alternatively, average RSD must be <15%.	At minimum, yearly or as necessary due to major instrument maintenance or continuing difficulties meeting the CCV requirements.	Re-analysis of out of control standards.	Copies of all raw data, mass calibration, tune, and Form VI.
Internal Standards	Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 40 ng/uL ea. Area must be -50% to +100% of the IS in the CCV. RT must be within ±0.06 RT units of the RT of the IS in the CCV.	Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 2.0 ng/ul ea. Area must be -50% to + 100% of the area of the IS in the CCV. RT must be within 0.5 minutes of RT in CCV.	All standards, blanks, matrix spikes, matrix spike duplicates, blank spikes, SRMs, and sample re-extracts.	Re-analyze any sample which IS areas or RTs are out of range.	
DFTPP Tuning Verification (50 ng)	Mass Abundance 51 30-60% of 198 68 <2% of 69 69 present 70 <2% of 69 127 40-60% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >1.0% of 198 441 present but < 443 442 40-100% of 198 443 17-23% of 442	Mass Abundance 51 30-60% of 198 68 <2% of 69 69 present 70 <2% of 69 127 40-60% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >1.0% of 198 441 present but < 443 442 40-100% of 198 443 17-23% of 442	Every 12 hours; CLP Criteria is used as allowed by the method.	Retune instrument to pass criteria.	Copy of DFTPP check with the file.
Continuing Calibration Verification	Mid-level standard every 12 hours. %D <20% for CCCs, all others <30.	1.0 ng/ul standard every 12 hours. %D for CCC<20, all others<30.	Every 12 hours.	Perform system maintenance, re-analyze CCV standard.	Copies of raw data, mass calibration, tune, and Form VII.
Method Blank	Analytes must be <MDL	All analytes <RL.	One method blank per 20 samples or each extraction batch of samples, whichever is more frequent.	Re-analyze blank. If still out of control, re-extract the entire batch of samples unless the offending analyte(s) are not detected in the associated samples.	Notation in instrument log and case narrative if applicable.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Surrogate Recovery	Upper and lower recovery limits determined by 3X standard deviation of at least twenty samples. To be performed at least semi-annually.	1-Fluoronaphthalene, Fluorene-d10, Pyrene-d10 surrogates are used. Limits are updated annually.	All samples, method blanks, blank spike, MS/MSD.	Re-extraction of the sample is required if any surrogate is out. All surrogates must be in control in the method blank otherwise all associated samples must be re-extracted..	Any out of control surrogates are to be documented in the instrument logbook and the job comments section or case narrative.
Blank Spike Recovery	Empirically derived from 20 or more BS. QC interval equals 3X standard deviation.	Empirically derived from 20 BS. QC interval equals 3X standard deviation.	One per batch of twenty.	Re-extract batch unless MS/MSD is perfect.	Narrate in case narrative.
MS/MSD Recovery & RPD	Empirically derived from 10 or more MS/MSD pairs. QC interval equals 3X standard deviation.	Empirically derived from 10 or more MS/MSD pairs. QC interval equals 3X standard deviation.	One per batch of twenty.	Re-extract batch unless blanks spike is perfect or documentable matrix effect is present.	Narrate problems in case narrative.

APPENDIX IX

Analytes Ammenable to Analysis by Method 8270C SIM

Current 8270 SIM PNA Target Analytes:

Compound	Primary Ion	Secondary Ion(s)
Naphthalene	128	129,127
2-Methylnaphthalene	142	141
Acenaphthylene	152	151,153
Acenaphthene	153	154,152
Fluorene	166	165,167
Phenanthrene	178	179,176
Anthracene	178	176,179
Carbazole	167	166
Fluoranthene	202	200
Pyrene	202	200,203
Benzo(a)anthracene	228	229,226
Chrysene	228	226,229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	274
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	274,277
1-Fluoronaphthalene	146	147,145
Fluorene-d10	176	174,177
Pyrene-d10	212	

APPENDIX X

Elution Order of the Compounds

Compound	Retention Time (min)
Naphthalene	8.71
2-Methylnaphthalene	10.57
Acenaphthylene	13.09
Acenaphthene	13.62
Fluorene	15.04
Phenanthrene	17.76
Anthracene	17.89
Carbazole	18.41
Fluoranthene	21.16
Pyrene	21.78
Benzo(a)anthracene	25.19
Chrysene	25.31
Benzo(b)fluoranthene	28.13
Benzo(k)fluoranthene	28.21
Benzo(a)pyrene	29.08
Indeno(1,2,3-cd)pyrene	33.00
Dibenzo(a,h)anthracene	33.06
Benzo(g,h,i)perylene	34.11
1-Fluoronaphthalene	8.70
Fluorene-d10	14.96
Pyrene-d10	21.74

APPENDIX XI

Semivolatile Internal Standards With Corresponding Analytes Assigned For Quantitation

Naphthalene-d8	Acenaphthene-d10
Naphthalene	Acenaphthylene
2-Methylnaphthalene	Acenaphthene
1-Fluoronaphthalene	Fluorene
	Fluorene-d10

Phenanthrene-d10	Chrysene-d12	Perylene-d12
Phenanthrene	Pyrene	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
Carbazole	Chrysene	Benzo(a)pyrene
Fluoranthene	Pyrene-d10	Indeno(1,2,3-cd)pyrene
		Dibenzo(a,h)anthracene
		Benzo(g,h,i)perylene

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8302

Title: HPLC Ordnance Data Review

Revision history:

Number	Date
0	07/31/95
1	01/23/98

Revised by: Monica Carr Date: 1/30/98
Monica Carr, Organic Division Manager

Approved by: Harry Romberg Date: 1-30-98
Harry Romberg, QC Officer

Approved by: Karen J Kotz Date: 1/26/98
Karen Kotz, Laboratory Director

UNCONTROLLED

Summary

The data reviewer will be working on reviewing data packages which have been completed by the HPLC analyst. The review will follow this procedure, utilizing reference materials attached to this procedure as well as client/project specific requirements (such as Maximum % RSD and CRDLs, if different from EPA PQLs), which will be provided by the HPLC Supervisor. All data review will be documented on the "Data Review Checklist" (attached to this procedure) and comments will be referenced to the Review Item and number. At the end of the review process, the reviewer will arrange the data package in the order specified here, with the completed "Data Review Checklist" and accompanying notes from the analyst(s) (if applicable) on the top of the package. This package will be given to the HPLC Supervisor for final review and narrative preparation.

List of Attachments

1. Laucks Testing Labs - Data Review Checklist [DRC] (HPLC - Ordnance Version)

Procedure

1. Initial Information

   A. Before beginning the review process, it is important to confirm with the HPLC Supervisor what method was used and what the associated QC limits are. Most of this information is printed on the following forms, however it must be confirmed that these QC limits are correct for the method/client/project. The levels for the following QC criteria should be confirmed:

Initial Calibrations:	Maximum %RSD	*(usually 20%, will be on Form V)*
Continuing Cals:	Maximum %D	*(usually 15%, will be on Form VII & ICV)*
Surrogates:	Acceptable Recovery Range *(will be on Form II)* and spiking levels *[100 ul of Surrogate Spike Soln. Residue levels for Water and Soil will be 2 ppm.]*	
Blank:	Maximum Allowable Concentration for confirmed compounds	
MS/MSD:	QC Limits for % Rec and % RPD *(on Form III)* and spiking levels *[100 ul of Spiking Solution. Water will be 4 ppb, Soil will be 133 ppb.]*	

   B. Laucks forms in the data package

	Laucks #	EPA Equivalent
QC Summary Data:		
"Surrogate Recovery Summary Report"	SURR GC	*Form II*
"MS/MSD Report" *and Blank Spikes/Duplicates*	BLKSPK-1	*Form III*
	BLKSPK-2	
	MS/MSD-1	
	MS/MSD-2	
"Method Blank Summary"	BLK SUMM	*Form IV*
Sample Data: *(and Raw QC Data)*		
"Organics Analysis Data Sheet"		*Form I*
"Compound Confirmation"	GC CONF	*Form X*

B. Laucks forms in the data package *(continued)*

	Laucks #	EPA Equivalent
Standards Data:		
"Initial Multi-point Calibration"	ICAL GC	*Form VI*
Cal. Factors:	Part 1	
RTs:	Part 2	
Peak Response (Areas):	Part 3	
Linear Regression:	Part 4	
"Initial Calibration Verification Worksheet"	ICV-1	
"Continuing Calibration Verification Worksheet"	CCV-1	*Form VII*
Calibration Factor:	Page 1	
Retention Times:	Page 2	

2. Overall Review

   A. Review Narrative already written by analyst to familiarize yourself with the project and to confirm what comments have already been written.

<u>QC Summary</u>

3. Form II ("Surrogate Recovery Summary Report")

   A. Confirm all sample and QC are present by comparing to the bench sheet.
   Check "Yes" or "No" on DRC #A1. Discrepancies must be noted on the DRC, referencing #A1.

   B. Check to make sure the recoveries for surrogates are within QC limits. Outliers will be marked with an "*". Comment on any that are outside limits or are not present (have been diluted out).
   Check "Yes" or "No" on DRC #A2 at completion of review. If any recoveries are outside QC Limits, these must be noted on the DRC, referencing #A2.

4. Form III ("MS/MSD Report" *and Blank Spikes/Duplicates* BLKSPK-1,2, MS/MSD-1,2)

   A. **BLKSPK-1 and/or MS/MSD-1** - Confirm the spike added against the normal levels of 4 ppb for water, 133 ppb for soil. If not the normal levels, confirm against the bench sheets and log books.

   B. **BLKSPK-1 and/or MS/MSD-1** - Check the amount found against Form I's (these are entered manually on Form III's) for the corresponding sample (check Lab ID against bench sheet). Check to see if any % Rec, and/or RPD's are out of control (will be indicated with an "*").
   Check "Yes" or "No" on DRC #A3 and #A4 at completion of review. The number and type (high or low) of out of controls must be noted on the DRC, referencing #A3 and/or #A4 followed by the Lab ID # where the out of controls occurred.

   C. **BLKSPK-2 and/or MS/MSD-2** - Check that the correct samples are associated with the blank spike and/or MS/MSD by confirming against the bench sheet.
   Check "Yes" or "No" on DRC #A5 at completion of review. Discrepancies must be noted on the DRC, referencing #A5.

5. Form IV ("Method Blank Summary")

   A. Check the blank name and corresponding lab ID numbers against the bench sheet.
   Check of "Yes" or "No" on DRC #A6. Discrepancies must be noted on the DRC, referencing #A6.

Standards Data

7. Form VI ("Initial Multi-point Calibration" ICAL HPLC

Cal. Factors:	Part 1
RTs:	Part 4
Peak Response (Areas):	Part 2
Linear Regression:	Part 3
Amount Summary:	Part 5

   A. **Part 1** - Check that % RSD is within 20% limits (will be marked with a "*" if outside limit). Also confirm that the lab file ID's match those on the Target Sequence for the ICAL.
   Check "Yes" or "No" on DRC #C2 at completion of review. Discrepancies must be noted on the DRC, referencing #C2.

8. "Initial Calibration Verification Worksheet" ICV-1

   A. There may be an ICV Form for the mid-point standard from the ICAL, but only if samples were analyzed directly after the ICAL, before another CCV. Check that there is an ICV associated with each analytical batch of samples reported here by checking date and time against Form IV ("Method Blank Summary"). If unclear as to which ICVs correspond to the samples, the Target Sequence should be referenced.
   Check "Yes" or "No" on DRC #C1 at completion of review. Discrepancies must be noted on the DRC, referencing #C1.

   B. Confirm that %D's are within limits (15%). They will be marked with a "*" if out.
   Discrepancies must be noted on the DRC, referencing #C1.

9. Form VII ("Continuing Calibration Verification Worksheet" CCV-1)

Calibration Factor:	Page 1
Retention Times:	Page 2

   A. **Page 1** - Check that % D is within 15% control limits. Confirm that all forms are included here by comparing against the Target Sequence.

   B. **Page 2** - Confirm Continuing Calibration RTs are within RT window (note: these will <u>not</u> be marked with a "*" if out of control).

   Check "Yes" or "No" on DRC #C4 at completion CCV of review. Discrepancies must be noted on the DRC, referencing #C4.

10. Chromatograms and Processed Files

   A. Confirm that chromatograms and processed files for all calibration standards and CCVs are present.
   Check "Yes" or "No" on DRC #C5 at the completion of review. Discrepancies must be noted on the DRC, referencing #C5.

Sample Data and **Raw QC Data**

12. Form I ("Organics Analysis Data Sheet")

A. Check dates received and dates of collection against the SDG Database Report, and date extracted against the extraction bench sheets. Confirm that the dates extracted and analyzed were within holding times.
   Extraction: 14 days for soils and 7 days for waters (measured from date of collection).
   Analysis: 40 days from extraction.
   Check "Yes" or "No" on DRC #B1 at the completion of review. Discrepancies must be noted on the DRC, referencing #B1.

B. Check sample size against the bench sheet - the initial sample (there are sometimes many dilutions) will always be a 1:2 dilution for water and soil. Confirm that the percent moisture is correct by comparing to the SDG report (for soils). Waters should always be 100% moisture.
   Check "Yes" or "No" on DRC #B2 at the completion of review. Discrepancies must be noted on the DRC, referencing #B2.

C. Check that all "hits" are correctly transferred to Form I from Form X (unless they are crossed out on Form X). In addition, check the retention times of the analyte to ensure that they are within the RT window. The retention times and RT windows are located on the compound confirmation sheet.
   Check "Yes" or "No" on DRC #B3 at the completion of review. Discrepancies must be noted on the DRC, referencing #B3.

D. Were all samples with analyte concentrations > the highest calibration standard diluted and reanalyzed? If so, were the diluted sample results linear with the last result that exceeded calibration (i.e. within 25%)?
   Check "Yes", "No" or "N/A" on DRC #B4 at completion of review. Discrepancies must be noted on the DRC, referencing #B4.

E. Check that the sample number on Form I matches that on the chromatogram.
   Check "Yes" or "No" on DRC #B5 at the completion of review. Discrepancies must be noted on the DRC, referencing #B5.

F. Check that flags are used correctly. Flags not referenced on the "Use of Data Qualifiers" Memo are detailed below.
   "D" These flags are used on diluted sample results, but are not used on the initial 1:2 dilution as this is a dilution of methanol with water to produce the appropriate sample matrix for HPLC analysis.
   "E" Anything above 1000 ppb extract concentration on Form X must be flagged with an "E" on Form I and re-diluted to attain an accurate analysis within the calibration curve.
   "X" This will be used for the most accurate "hit" from all the dilutions. Only one "X" for each sample/analyte should occur throughout the dilutions, as there is only one "most accurate" hit.
   "Z" A "Z" flag on sample report forms indicates coelution has occurred between two or more target analytes on the confirmation CN column. For this reason, quantitative confirmation is not possible.
   Discrepancies must be noted on the DRC, referencing #B6.

13. Form X ("Compound Confirmation")

A. Check that any %D over 25% is "P" flagged on Form I (note that blank and matrix spikes are not confirmed and %D's reported should be crossed out for blank spikes).
Check "Yes" or "No" on DRC #B6 at the completion of review of data qualifiers. Discrepancies must be noted on the DRC, referencing #B6.

B Check the retention time windows located on the the compound confirmation form, with the copy of the retention time window study located in the Bench Sheet section of the data package.
Check "Yes" or "No" on DRC #B7 at the completion of review of data qualifiers. Discrepancies must be noted on the DRC, referencing #B7.

At the end of the Sample Data and Raw QC review, remove all Form Xs and place them at the end of the Standards Data set.

14. Final Review

A. Were recoveries for the SRM (if required by client/project) within QC Limits?
Check "Yes", "No" or "N/A" on DRC #D2 at completion of review. Discrepancies must be noted on the DRC, referencing #D2.

B. Are all nonconformances included and noted on the DRC?
Check "Yes" or "No" on DRC #D1 at completion of review. Discrepancies must be noted on the DRC, referencing #D1.

17. Order of Final Packet - All in numerical and chronological order and in order by column
Check "Yes" or "No" on DRC #D3 at review completion. Note discrepancies referencing #D3.

1. QC Summary
Forms II through IV

2. Sample Data
For each sample, the following packet:
All Form I's, then chromatograms and integration reports *(C18 first, then CN column)*

3. Standard Data
Form VI's (Parts 1,2,3, and 4) *(C18 column first, then CN column on each)*
ICV-1 (Initial Calibration Verification Sheets)
Form VII's (Pages 1 and 2) *(C18 column first, then CN column on each)*
Form X's *(samples first, followed by QC samples)*
Raw Standards Data, *(compound confirmation, chromatograms and integration reports)* including:
Multi-point calibrations *(C18 column, then CN column)*
All CCVs and ICVs in chronological order
MDL Standard
I Blanks

4. Raw QC Data *(Method Blanks, Blank Spikes/Blank Spike Duplcts, MS/MSDs, Control charts.)*
   For each sample, the following packet:
   All Form I's, then chromatograms and integration reports *(C18 first, then CN column)*

5. Bench Sheets
   Including: SDG Report, Copy of instrument logbook, Extraction bench sheets, Logbooks for surrogates/spikes (referenced on bench sheet), Logbooks for standards, RT Study.

Attachment 1
Data Review Checklist

Laucks Testing Labs · Data Review Checklist
Ordnance 8330 - HPLC Department

Work Order Number:	
Analysis Batch Dates:	
Method:	SDG #:

Review Item	Yes	No	N/A	2nd Review
A. QC Summary 1. Are all required QC results present?				
2. Do surrogate recoveries meet QC limits?				
3. Are blank spike/blank spike duplicate results correct and do they meet QC %Rec and RPD limits?				
4. Are MS/MSD results correct and do they meet QC %Recovery & RPD limits?				
5. Are correct samples associated with the blank spike(s) and/or MS/MSDs?				
6. Are all blanks reported?				
7. Is MDL recovery within QC limits?				
B. Sample Data and Raw QC Data 1. Were samples extracted and analyzed within holding times?				
2. Are sample sizes and % moisture correct?				
3. Are all hits recorded correctly?				
4. Were samples diluted as required and were dilutions linear (within 25% of last value)?				
5. Were sample ID's checked?				
6. Were data qualifier flags used correctly?				
7. Were the retention time windows on the compound confirmation form recorded properly? Review RT study located in Bench Sheet section.				
C. Standards Data 1. Is there an ICV run every day and are all %Ds within QC limits?				
2. Are calibration %RSDs within QC limits?				
3. Are all ICVs and CCVs recorded and are RT %Ds within QC control limits?				
4. Are CCV %Ds and RTs within QC limits?				
5. Is all raw data present?				
D. Final Review 1. Are all nonconformances listed?				
2. Are recoveries for the SRM within limits?				
3. Are all components of the data package present and in correct order?				

Laucks Testing Labs Data Review Checklist
Ordnance 8330 - HPLC Department

Work Order Number:	
Analysis Batch Dates:	
Method:	SDG #:

Comments on any "No" responses:

Data Reviewer: ______________________ **Date:** ______________

2nd Level Reviewer: ______________________ **Date:** ______________

LAUCKS TESTING LABORATORIES INC.

Seattle, Washington

SOP #:LTL-8330

Determination of Nitroaromatics and Nitramines by SW-846 Method 8330

Revision history:

Number	Date
1	06/19/95
2	01/11/96
3	04/10/96
4	04/15/96
5	07/09/96
6	12/18/97
7	4/30/98
8	10/28/98
9	01/25/99

UNCONTROLLED

Revised by: Monica Carr
Monica Carr, Organic Division Manager
Date: 1/25/99

Approved by: Harry Romberg
Harry Romberg, QA Officer
Date: 1-25-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 1-25-99

TABLE OF CONTENTS

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the trace analysis of ordnance compounds in water, soil, and sediment samples by high performance liquid chromatography. The concentrated water sample extracts are diluted 1:1 (v/v) with reagent grade water prior to analysis. The sample extracts are analyzed using a C18 (octadecyl) reverse phase column, and target analyte concentrations are measured at either 254 nm or 210 nm using a UV detector. All positive measurements observed on the C18 column are confirmed by a second analysis which uses a CN (cyano) column. The C18 column is considered the primary column and is used for quantitation of all target analytes.

1.1.2 This method is used to determine part per billion levels of the ordnance analytes listed below:

Compound	Acronym
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Methyl-2,4,6-trinitrophenylnitramine	Tetryl
Nitrobenzene	NB
2,4,6-Trinitrotoluene	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	4-Am-DNT
2-Amino-4,6-dinitrotoluene	2-Am-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

6 Additional Compounds
(Separate Analysis at 254 nm)

Compound	Acronym
2,4-Diamino-6-nitrotoluene	2,4-DA-6-NT
2,6-Diamino-4-nitrotoluene	2,6-DA-4-NT
3,5-Dinitroaniline	3,5-DNA
1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine	MNX
1,3,5-Trinitroso-1,3,5-hexahydrotriazine	TNX
2,2',6,6'-Tetranitro-4,4'-azoxytoluene	4,4'-TN-AZOXY

PETN/NG Compounds
(Separate Analysis at 210 nm)

Compound	Acronym
Nitroglycerin	NG
Pentaerythritoltetranitrate	PETN

1.1.3 Aqueous samples of higher concentration can be directly analyzed by diluting 1:1 (v/v) with methanol or acetonitrile, filtering, separating on a C18 reverse phase column, and determined at either 254 nm or 210 nm, and confirmed on a CN column.

1.1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of high pressure liquid chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations from the published version of method SW 8330 which are followed as standard operating procedure in the performance of this method at Laucks.

1. Single injections of calibration standards are analyzed rather than triplicate injections in a random order. It has been determined at Laucks that single injections of these standards yield acceptable calibration and linearity data as evidenced by the calculated percent RSDs used to evaluate the initial calibration data.

2. Method SW 8330 specifies that all analyses are to be performed using a mobile phase which consists of a 50:50 mixture of methanol/water under

isocratic conditions. Laucks employs a gradient elution program (detailed in Section IV) in order to improve the separation of the target analytes on the CN column.

3. According to Method 8330, all working standards are to be prepared daily in a methanol/calcium chloride solution. The practice at Laucks for 8330 and the 6 additional compounds is to prepare working standards using acetonitrile and diluting 50/50 with water just prior to analysis. NG/PETN are prepared in a 50/50 mixture of acetonitrile and water. These working solutions have been demonstrated to be stable for at least 6 months. The stability of target analyte responses in the working solutions in use (especially that of tetryl) is used to determine whether new solutions should be prepared.

4. Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. Laucks has experienced erratic recovery of tetryl from spiked sample extracts.

5. The confirmation column demonstrates full or partial coelution for the following target analyte pairs: 1,3-dinitrobenzene and 1,3,5-trinitrobenzene; 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene; and 2,4-dinitrotoluene, 2,6-dinitrotoluene and 2,4,6-trinitrotoluene. Therefore, positive confirmation can not be made when two or more co-eluting peaks have been tentatively identified on the primary column.

6. At the time of this writing, analysis of the 6 additional compounds (not including PETN/NG) combined with the 14 ordnance compounds results in the co-elution of 2,6-DA-4-NT and HMX; and 3,5-DNA and tetryl on the C18 column using the 8330 method. However, tetryl and HMX do not co-elute with any attenuation compound on the CN column using the 8330 method. These two compounds (HMX and tetry) do not co-elute with any of the attenuation compounds on the primary colum nor confirmation column using the attenuation method.

7. Although the compounds NG and PETN are not listed in SW 846 Method 8330, Laucks has found that adequate recovery of these compounds can be achieved using this method by modifying the wavelength from 210 nm to 254 nm.

8. Picric and picramic acids exhibit substantial peak shift and may elute near or co-elute with HMX on the C18 column using the 8330 method. However, no attenuation compounds, nor NG/PETN coleute on the primary (PAH)

column using the picric/picramic method. The buffer is used in this method in order to stablize these compounds.

1.2.2 In order to attain lab-wide consistency among staff members for decision-making processes with regard to laboratory anomalies, several common items have been addressed in this SOP. Any occurrences which are not covered in this SOP should be discussed with the supervisor, prior to implementing a solution.

1.2.3 One example is the determination of potential carry-over in sample analyses. Any samples analyzed subsequent to a high level sample (which is defined by yielding one or more target analytes above the calibration range) should be thoroughly examined for potential carry-over of the same target analyte(s). Corrective action in the form of reanalysis for possible carry-over should be performed and documented in the narrative.

1.2.4 Another example is the review of all sample chromatograms for analytes which may not show up on the sample quantitation report due to data system error or retention time shift. All peaks should be examined and evaluated based on the retention times and sample concentration in order to prevent reporting false negatives.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ±2°C. According to guidelines listed in SW-846 for the extraction of semivolatile compounds, water samples must be extracted within 7 days of collection, and soil samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

1.3.2 Although not a routine sampling practice, a soil holding time study performed by the U.S. Army Cold Regions Research and Engineering Laboratory recommends a maximum holding time of eight weeks when samples are frozen.

1.3.3 A similar study performed for water samples recommends a maximum holding time of 50 days for relatively sterile samples which are refrigerated. Surface waters, or samples likely to have significant microbial activity, may suffer significant losses of nitroaromatic compounds (particularly trinitrobenzene and trinitrotoluene) within 1-2 days, even under refrigeration. If microbial activity is suspected, water samples should be extracted as soon after collection as is practical.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CF	Calibration factor. The ratio of peak response to nanograms injected. This term is defined in the same way in both the CLP contract and SW-846.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	An instrument blank is solvent containing the method surrogates and is injected into the instrument to monitor for carryover between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined at Laucks.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW-846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts, and terminated with a standard injection. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention time window. The ± value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

Primary Column:	C18 (octadecyl) reverse phase HPLC column, 15 cm x 4.6 mm, 5 µm particle size, (Rainin Microsorb or equivalent).

Secondary Column:	CN (cyano) reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm particle size, (Supelcosil LC-CN or equivalent). C8 and CN reverse phase HPLC columns in series, 10cm x 3.9mm, 4µm particle size, (Waters Nova- Pak or equivalent). These are used in the for the analysis of the 6 additional compounds.
Mobile Phase:	Methanol (EM Science brand high purity solvent or equivalent). Reagent water (Modulab Polisher HPLC grade water or equivalent).
UV Detector:	254 nm (210 nm for NG and PETN)
HPLC System:	Rainin HPLC system - HPXL solvent delivery system capable of achieving 4000 psi. 50 µL sample loop. Knauer variable wavelength UV detector. Dynamax automatic sample injector. Digital integrator: EZChrom.
Waters System:	Waters 712 WISP Sample Processor or equivalent. 50 µL sample loop or equivalent. Waters 486 tunable absorbance detector or equivalent. Waters 600E Multisolvent delivery system or equivalent. Digital Integrator: EZChrom.
Hewlett Packard System:	HP 1313A Autosampler. HP G1311A QuatPump. HP G1322A On-line degasser. HP G1316A Thermostat column compartment. HP 1314A Variable wavelength detector. Digital Integrator: EZChrom.

2.1.1 Column Temperature Control:

2.1.2 Column temperature is controlled through the use of a column heater which is maintained at a temperature of 25°C.

2.2 Standards

2.2.1 Target Analyte Stock Solution

2.2.1.1 The stock solution used contains the 14 standard compounds in the mix at a concentration of 1000 µg/mL and is generally purchased from AccuStandard Inc. (25 Science Park, New Haven CT 06511). Equivalent solutions from this or other vendors are also acceptable.

2.2.1.2 The NG and PETN stock solutions are purchased individually from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The NG solution comes in 1 mL ampules at 4000 µg/mL in ethanol. The PETN solution comes in 1 mL ampules at 1000 µg/mL in methanol. Equivalent solutions from this or other vendors are also acceptable.

2.2.1.3 The 3,5-Dinitroaniline and 1,3,5-Trinitroso-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from the U.S. Army Environmental Center (Aberdeen Proving Ground, MD 21010). The 2,4-Diamino-6-nitrotoluene, 2,6-Diamino-4-nitrotoluene and 2,2',6,6'-Tetranitro-4,4'-azoxytoluene standards are made from standard analytical reference materials obtained from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The 1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from Stanford Research.

2.2.1.4 Calibration standard, surrogate stock and IBLK surrogate solutions are detailed in Appendix I.

2.2.2 Surrogate Stock Solution

2.2.2.1 The surrogate stock solution is received in a methanolic solution at a concentration of 1000 µg/mL. The surrogate can also be prepared by weighing 100 mg of 1,2-dinitrobenzene into a 100 mL volumetric flask and diluting to volume with methanol to yield a concentration of 1000 µg/mL.

2.2.3 IBLK Working Solution

2.2.3.1 The working instrument blank solution contains the surrogate compound only at a concentration of 2.0 µg/mL. Prepare this solution by adding 100 µL of the 1000 µg/mL surrogate stock solution to a 50 mL volumetric flask. Dilute to volume so that the final solvent concentration is methanol/water at a ratio of 1:1.

2.2.4 Working Calibration Standards

2.2.4.1 Prepare the working calibration standards for the 14 standard analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis. These 14 analytes are combined into one working solution, since they do not co-elute on the primary column.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentration (µg /mL)
Standard #6	A	0.20	10	20.0
	B	0.20		
Standard #5	A	0.1	10	10.0
	B	0.1		
Standard #4	C	1.0	10	2.0
Standard #3	D	1.0	10	1.0
Standard #2	E	1.0	10	0.2
Standard #1	F	1.0	10	0.1

Source Solution	Concentration (µg /mL)
A = 14 Component Mix	1000
B = Surrogate Stock Solution	1000
C = Standard #6	20
D = Standard #5	10
E = Standard #4	2.0
F = Standard #3	1.0

2.2.4.2 Prepare the working calibration standards for the 6 additional analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations (µg /mL)
Standard #6	A	2.0	10	20.0
	B	0.2		
	C	0.2		
	D	0.2		
	E	0.2		
	F	2.0		
	G	0.2		
Standard #5	H	5.0	10	10.0
Standard #4	H	1.0	10	2.0
Standard #3	I	1.0	10	1.0
Standard #2	J	1.0	10	0.2
Standard #1	K	1.0	10	0.1

Source Solution	Concentration (µg/mL)
A = MNX	100
B = TNX	1000
C = 3,5-DNA	1000
D = 2,6-DA-4-NT	1000
E = 2,4-DA-6-NT	1000
F = 4,4'-TN-AZOXY	100
G = 1,2-DNB	1000
H = Standard 6	20.0
I = Standard 5	10.0
J = Standard 4	2.0
K = Standard 3	1.0

2.2.4.3 Prepare the working calibration standards for PETN/NG in the following manner. All solutions are prepared in a 1:1 acetonitrile/water mixture.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations NG:PETN:1,2-DNB (µg /mL)
Standard #5	A	0.05	10	20.0
	B	0.10	10	10.0
	C	0.10	10	10.0
Standard #4	D	5.0	10	10.0, 5.0, 5.0
Standard #3	E	5.0	10	5.0, 2.5, 2.5
Standard #2	F	2.0	10	1.0, 0.5, 0.5
Standard #1	G	5.0	10.0	0.5, 0.25, 0.25

Source Solution	Concentration (µg/mL)
A = Nitroglycerin	4000
B = PETN Solution	1000
C = Surrogate Stock Solution	1000
D = Standard 5	As Above
E = Standard 4	As Above
F = Standard 3	As Above
G = Standard 2	As Above

2.2.4.4 Note: Store all standard and surrogate solutions in amber glass vials with screw caps and Teflon-lined septa. Minimize headspace in these vials and store them in a refrigerator kept at 4°C ±2°C. Allow standards to come to room temperature prior to use.

2.2.4.5 Stock solutions may be used for up to one year, and working solutions are good for at least six months, or the expiration date of the parent standard, whichever is sooner.

2.2.4.6 All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation standards.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 All compounds analyzed by this method are used either in the manufacture of explosives or are the degradation products of these compounds. When making stock solutions for calibration, treat each explosive compound with caution.

3.1.3 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.4 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.5 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 Out of date standards and sample extracts are disposed of in the designated organic waste container located in the organic preparation area.

3.2.2 HPLC liquid waste is disposed of by pouring into the designated organic waste container located in the solvent storage area.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Current C18 Column Conditions for the 14 Compounds:

HPLC3		HPLC4	
Pump A Solvent:	Methanol	Solvent:	Methanol/Water
Pump B Solvent:	Water	Flow:	0-24 min = 0.5 mL/min
Flow:	1.0 mL/minute:		24-35 min = 0.8 mL/min
Gradient:	Linear		35-40 min = 0.5 mL/min
Run Time:	60 minutes	Run Time:	40 minutes
Acquisition Time:	60 minutes	Acq. Time:	35 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

Gradient Profile (HPLC3)		Run Time Profile
Initial %A:	40	
Initial %B:	60	
Hold Time:	5 minutes	0 - 5 minutes
Gradient 1 %A:	60	
Gradient 1 %B:	40	
Ramp Time 1:	15 minutes	
Hold Time 1 :	5 minutes	5 - 20 minutes
Gradient 2 %A:	95	
Gradient 2 %B:	05	
Ramp Time 2:	10 minutes	20 - 30 minutes
Hold Time 2:	5 minutes	30 - 35 minutes
Gradient 3 %A:	40	
Gradient 3 %B:	60	
Ramp Time 3:	5 minutes	35 - 40 minutes
Hold Time 3:	20 minutes	40 - 60 minutes (Column Equilibration)

4.1.2 Current CN Column Conditions for the 14 Compounds:

HPLC2 File #6:		
Pump A Solvent:	Methanol	
Pump B Solvent:	Water	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	50 minutes	
Acquisition Time:	25 minutes	
Initial %A	25	
Initial %B	75	
Gradient 1 %A:	45	
Gradient 1 %B:	55	
Ramp Time 1:	2 minutes	0 - 2 minutes
Hold Time 1 :	3 minutes	2 - 5 minutes
Gradient 2 %A:	60	
Gradient 2 %B:	40	
Ramp Time 2:	5 minutes	5 - 10 minutes
Hold Time 2 :	8 minutes	10 - 18 minutes
Gradient 3 %A:	25	
Gradient 3 %B:	75	
Ramp Time 3:	7 minutes	18 - 25 minutes
Hold Time 3 :	25 minutes	25 - 50 minutes

4.1.3 Current C18 Column Conditions for the 6 Additional Compounds:

Gradient Profile (HPLC3)		**Run Time Profile**
HPLC3 File:	EXTRAS	
Pump A Solvent:	Water	
Pump B Solvent:	Methanol	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	53 minutes	
Acquisition Time:	30 minutes	
Initial %A	90	
Initial %B	10	
Gradient 1 %A:	70	
Gradient 1 %B:	30	
Ramp Time 1:	5 minutes	0 - 5 minutes
Hold Time 1 :	0 minutes	
Gradient 2 %A:	50	
Gradient 2 %B:	50	
Ramp Time 2:	3 minutes	5 - 8 minutes
Hold Time 2 :	7 minutes	8 - 15 minutes
Gradient 3 %A:	26	
Gradient 3 %B:	74	
Ramp Time 3:	2 minutes	15 - 17 minutes
Hold Time 3:	13 minutes	17 - 30 minutes
Gradient 4 %A:	90	
Gradient 4 %B:	10	
Ramp Time 4:	2 minutes	30 - 32 minutes
Hold Time 4:	21 minutes	32 - 53 minutes (Column Equilibration)

4.1.4 Current C8-CN in Series for the 6 Additional Compound Confirmation:

Time	Flow	Methanol	Water
	1.0	15	85
2.4	1.0	15	85
7.0	1.0	40	60
10.0	1.0	50	50
17.0	1.0	50	50
20.0	1.0	80	20
21.0	0	15	85

4.1.5 Current Column Conditions for NG/PETN:

<u>C18</u>		<u>CN</u>	
Pump A Solvent:	Methanol	Pump A Solvent:	Methanol
Pump B Solvent:	Water	Pump B Solvent	Water
Flow:	0.6 mL/minute:	Flow:	1.2 mL/minute
Gradient:	Linear (1:1)	Gradient:	Linear (1:1)
Run Time:	30 minutes	Run Time:	20 minutes
Acquisition Time:	30 minutes	Acq. Time:	20 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on determination of detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies are performed on both columns.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.4 Retention Time Windows

4.4.1 It is necessary to establish retention time windows for the method by analyzing standards for all target analytes over at least a 24-hour period. A 24-hour time period may be deemed more appropriate for this analysis because the instrument is not always operated for longer periods when analyzing sample extracts. At least 5 standards must be injected during the 24-hour time period, at about equal time intervals. These standards should be interspersed with real sample extracts in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds and compute the sample (n-1) standard deviations of all the retention times.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made or whenever the chromatographic method is modified.

4.4.3 In some cases, the retention time window may be modified in order to take into account any pattern shifting. This shift is acknowledged by observation of the surrogate peak behavior and the surrounding CCV standards. If retention time shift has occurred and the possibility of misidentifying peaks exists, then the sample is reanalyzed bracketed by in-control CCV standards.

4.4.4 See the Laucks SOP on determination of retention time windows for more specifics on their determination and use.

4.5 Initial Multi-Point Calibration

4.5.1 Analyze standard solutions (including the surrogate solution) using at least 5 different concentration levels. The lowest concentration defines the reporting limit. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. Criteria for evaluating these standards are detailed in Section VI.

4.6 Initial Calibration Verification

4.6.1 At the beginning of an analysis sequence, analyze the mid-point calibration standard (STD 3). The computed calibration factor (CF) or concentration measurement must meet the criteria detailed in Section VI.

4.6.2 Since the retention time windows which were established by the retention time study are relative and not absolute, the windows are anchored by the ICV. This allows the retention times from the ICV to become the mid-point of the retention time windows.

4.7 Instrument Blank

4.7.1 After the analysis of Standard 5, an instrument blank (IBLK) is analyzed. This is to verify that there is no carryover between sample injections. Evaluation criteria are detailed in Section VI.

4.7.2 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK. This IBLK analysis is used only to make a judgment as to the possibility of carryover into the sample analysis immediately following the IBLK. Evaluation criteria are detailed in Section VI.

4.8 Continuing Calibration Verification

4.8.1 A mid-range calibration standard (STD 3) is analyzed after every ten sample injections. In addition, this standard must be the last injection made in the analysis sequence. Evaluation criteria are detailed in Section VI.

4.9 Sample Analysis

4.9.1 Analysis sequence

See Appendix II for a detailed analysis injection sequence.

4.9.2 Compound Identification

4.9.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its retention time window on a second column. There are several analyte co-elutions which occur on the secondary column. In instances where target analytes that have been tentatively identified on the C18 column co-elute on the CN column, positive confirmation can not be made. All analyte quantitations come from the C18 column. The CN column is typically not used for quantitation purposes. The responses of the eluting compounds are summed and reported as one peak. When the presence of these compounds are indicated on the C18 column, CN column concentration values can not be accurately determined due to the coeluting peaks and the sample results are flagged appropriately. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria detailed in Section VI are strictly adhered to. Due to constraints of the software, the RT of the co-eluting compounds is determined by assigning the RT to the highest point within a give window. For the co-eluting compounds that may have a slight but not complete separation, such as: 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene, this determination of RT can result in RT windows which exceed those identified by the standard (due to the changes in peak heights with concentration values). In order to avoid potential exceedance of RT windows, and in order to accurately identify these co-eluting target analytes, the retention time windows may be administratively set for the co-eluting compounds.

4.9.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes. If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

4.9.3 Compound Quantification

4.9.3.1 Target compound concentrations are calculated using the following equations:

4.9.3.2 Aqueous samples

The external standard equation, as expressed in SW-846 is

$$Concentration(\mu g / L) = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times V_s}$$

where:

A_x	= Response for the analyte in the extract, in area or height units.
A	= Amount of standard injected (in nanograms).
A_s	= Response for the external standard, same units as A_x.
V_i	= Volume of extract injected, µL.
D	= Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if an extract is diluted 10 µL to 1000 µL and subsequently diluted an additional 10 µL to 1000 µL, the expression is: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t	= Volume of total extract, µL.
V_s	= Initial sample size, mL.

4.9.3.3 In routine use at Laucks, the equation reduces as follows.

4.9.3.4 First, CF is used directly in the equation. Since Cf = A_s/A, this substitution is made. Next, since Laucks routinely measures all final extract volumes in mL, a conversion factor for µL to mL must be made in the numerator of the expression - i.e., $V_t = 1000 * mL$. Finally, the sample preparation process is represented as the algebraic ratio of initial sample size to final extract volume.

4.9.3.5 The equation then becomes

$$Concentration(\mu g / L) = \frac{1000 \times A_x \times D}{CF \times Vi \times (Vs / Vi)}$$

4.9.3.6 This expression is completely equivalent to the SW-846 equation, yielding the same final result. To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/L * 0.001

4.9.3.7 Non-aqueous samples

4.9.3.8 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Concentration(\mu g / kg) = \frac{1000 \times A_x \times D}{CF \times V_i \times (W / V_t) \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

5. Reports

5.1 Data Packet Organization

See Appendix III for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Data Qualifying Flags

5.3.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U:	The analyte of interest was not detected, to the limit of detection indicated.
B:	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J:	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D:	The value reported is derived from the analysis of a diluted sample or sample extract.
P:	When a dual column technique is employed, this flag indicates that calculated results from the two columns differ by more than 25 percent.
E:	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
X:	The sample has been analyzed at several dilutions. The value reported has been determined to be the most appropriate quantitative value.
Z:	The value reported is from the C18 column only. Due to coelution with another target analyte on the CN column, the compound could not be quantitatively confirmed.

- Due to software constraints, the percent difference will also be calculated when quantitative confirmation is not possible on the confirmation column due to co-elution. When this happens, the concentration value of the confirmation column used to determine the %D is calculated as the sum of the co-eluting compounds and reported as one peak.

6. Quality Control

See the Requirements and Corrective Actions table in Appendix IV for additional information.

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 The initial calibration is evaluated by calculating the %RSD of the calibration factors from the five linearity standards.

6.1.1.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

6.1.1.3 This %RSD method assumes a linear response with the calibration curve passing through the origin.

6.1.1.4 The calculated CFs are tabulated and the %RSDs calculated. All compounds must have a %RSD of 20% to meet the method criteria.

6.1.2 Corrective action

6.1.2.1 If the %RSD criteria of 20% are not met, the out of control standard should be reanalyzed. If the curve is still out of control, determine the cause of failure and correct. Recalibrate the instrument and reanalyze any samples associated with the out of control curve.

6.2 Initial Calibration Verification

6.2.1 Criteria

6.2.1.1 Using the average CF calculation technique, compute the CFs for each compound. The calibration factors for the ICV standard are compared to the mean CFs for the initial multi-point calibration. The percent difference for these calibration factors is calculated as follows:

$$\% Difference = \frac{CF_a - CF_i}{CF_a} \times 100$$

where:
CF_a = .Average CF from the initial multi-point calibration
CF_i = Calibration Factor of the calibration verification standard.

6.2.1.2 The %D cannot exceed 15% for any target analytes or surrogates.

6.2.1.3 The mid-point for the RT window of each compound is updated using the ICV RTs.

6.2.2 Corrective action

6.2.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Determine the cause of the ICV failure and correct. Reanalyze the ICV and if it is still out of control, a new calibration curve must be analyzed.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 A CCV standard is analyzed singly for every 10 injections, and after the last sample of the sequence. The CF for each compound is calculated and the percent difference is calculated as shown above.

6.3.1.2 The %D results cannot exceed 15% for any target analytes or surrogates.

6.3.1.3 The retention times for all target analytes must fall within the RT windows established by the ICV.

6.3.2 Corrective action

6.3.2.1 Determine the cause of failure and correct. Reanalyze the calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit in the associated samples.

6.4 Instrument Blank

6.4.1 Criteria

6.4.1.1 There must be no target analyte levels above the reporting limit in the initial IBLK.

6.4.2 Corrective action

6.4.2.1 If the initial IBLK contains measurable levels of target analytes, the system is out of control. The source of contamination must be identified and corrected.

6.4.2.2 IBLKs are used to monitor for possible carryover in high concentration extracts. Those IBLKs optionally placed into the sequence following suspected high concentration extracts are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be reanalyzed if there is a detectable amount of the analyte found in the IBLK.

6.5 Method Blank

6.5.1 Criteria

6.5.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or at least one blank for every 20 samples, whichever is more frequent. Analytes of interest should not be detected at levels greater than the current reporting limit in the method blank sample. If any analytes are detected above the reporting limit, corrective action must be taken.

6.5.2 Corrective action

6.5.2.1 Reanalyze the blank and check calculations. If it is still out of control, re-extract the entire batch of samples unless the analyte(s) present in the method blank are not present in the associated samples. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

6.6 Blank Spike or QC Check Sample (LCS)

6.6.1 Criteria

6.6.1.1 A blank spike follows the same protocol as the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. In addition, this spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. A method blank with added analytes is a blank spike. Blank spike percent recovery control limits are detailed in the laboratory QC database. These control limits are updated periodically.

6.6.2 Corrective action

6.6.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re-extraction and reanalysis would be triggered by any analytes falling outside of control limits in the blank spike sample unless all sample surrogate recoveries and MS/MSD spike recoveries are in control.

6.7 Matrix Spike

6.7.1 Criteria

6.7.1.1 A sample is chosen at random (unless designated by project-specific requirements) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This frequency may be changed on a project specific basis. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. This spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:
SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.7.1.2 The current recovery criteria are detailed in the Laucks quality control database. These control limits are updated periodically.

6.7.2 Corrective action

6.7.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.8 Matrix Spike Duplicate

6.8.1 Criteria

6.8.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2)/2} \times 100$$

where:
S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.8.1.2 The current RPD control limits are detailed in the Laucks quality control database. These control limits are updated periodically.

6.8.2 Corrective action

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.9 Surrogate Recovery

6.9.1 Criteria

6.9.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:
S_m = Concentration of surrogate measured in sample.
S_a = Concentration of surrogate added.

6.9.1.2 Detailed surrogate recovery control limits are detailed in the Laucks quality control database. These control limits are updated periodically.

6.9.2 Corrective Action

6.9.2.1 Reanalyze the sample. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recoveries since non-GC/MS methods cannot separate co-eluting interferents. In instances where high surrogate recoveries are attributable to matrix effects, no corrective action is taken. However, if elevated surrogate recoveries are attributable to preparation error, re-extraction and reanalysis is performed.

6.9.2.2 Check calculations for possible error. Re-extract the sample if surrogate recovery is less than the lower control limit. If a poor injection is suspected, reanalyze the sample.

6.9.2.3 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8000B, "Gas Chromatography," Revision 1, July 1992, U.S.E.P.A.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, 3rd Update, Method 8330, "Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)," Revision 0, November 1992, U.S.E.P.A.

40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984, U.S.E.P.A.

Special Report 93-11, "Experimental Assessment of Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Soil," Clarence L. Grant et al, June 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

Special Report 93-24, "Evaluation of Pre-extraction Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Water," Clarence L. Grant et al, August 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

APPENDIX I

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
HMX	0.1	0.2	1.0	2.0	10.0
RDX	0.1	0.2	1.0	2.0	10.0
1,3,5-TNB	0.1	0.2	1.0	2.0	10.0
1,3-DNB	0.1	0.2	1.0	2.0	10.0
Tetryl	0.1	0.2	1.0	2.0	10.0
NB	0.1	0.2	1.0	2.0	10.0
2,4,6-TNT	0.1	0.2	1.0	2.0	10.0
4-Am-DNT	0.1	0.2	1.0	2.0	10.0
2-Am-DNT	0.1	0.2	1.0	2.0	10.0
2,4-DNT	0.1	0.2	1.0	2.0	10.0
2,6-DNT	0.1	0.2	1.0	2.0	10.0
2-NT	0.1	0.2	1.0	2.0	10.0
3-NT	0.1	0.2	1.0	2.0	10.0
4-NT	0.1	0.2	1.0	2.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.1	0.2	1.0	2.0	10.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
TNX	0.1	0.2	1.0	2.0	10.0
2,6-DA-4-NT	0.1	0.2	1.0	2.0	10.0
2,4-DA-4-NT	0.1	0.2	1.0	2.0	10.0
MNX	0.1	0.2	1.0	2.0	10.0
3,5-DNA	0.1	0.2	1.0	2.0	10.0
4,4'-TN-AZOXY	0.1	0.2	1.0	2.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.1	0.2	1.0	2.0	10.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
NG	0.5	1.0	5.0	10.0	20.0
PETN	0.25	0.5	2.5	5.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.25	0.5	2.5	5.0	10.0

Surrogate Stock Solution

Compound	Concentration
1,2-Dinitrobenzene	1000 µg/mL

IBLK Solution

Compound	Concentration
1,2-Dinitrobenzene	2.0 µg/mL

APPENDIX II

Analysis Sequence

Injection **Sample**

1 Standard #1
2 Standard #2
3 Standard #3 (ICV Standard)
4 Standard #4
5 Standard #5
6 IBLK
7 ICV
injections (8-16)

17 IBLK
18 CCV Standard
19 injections (19-27)
28 BLK
29 CCV Standard
injections (total of 10 injections between CCVs)

last IBLK
last CCV Standard

APPENDIX III

Data Packet Order List

I. QC SUMMARY

Surrogate Recovery Summary Report
Blank Spike Report
MS/MSD Report
Method Blank Summary

II. SAMPLE DATA:

Organic Analysis Data Sheet
Sample Confirmation Worksheet
Chromatogram, primary column
Quantitation Report, primary column
Chromatogram, secondary column
Quantitation Report, secondary column

III. STANDARD DATA:

Linearity Report
ICAL Data
ICAL Response, ICAL std concentrations
ICV reports
CCV reports
Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

Method Blank
Chromatograms
Quantitation Report

Blank Spike
Chromatograms,
Quantitation Report

Matrix Spike
Chromatograms
Quantitation Report

Matrix Spike Duplicate
Chromatograms
Quantitation Report

V. Bench Sheets

SDG Report
Extraction Bench Sheets
Injection Sequence (logbook copy)
Miscellaneous Work Sheets. (%TS, calcs, HTVRs)
Standards Logs

VI. Reject Data:

Data not used to support reported sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Initial Calibration	Minimum of 5 levels.	Minimum of 5 levels. Must demonstrate an RSD of <20%.	As necessary due to major instrument maintenance or difficulties meeting the CCV requirements.	Reanalyze out of control standard. If still out, determine cause of curve failure and correct. Re-analyze curve and any samples analyzed against curve.	Target forms and raw data.
Initial Calibration Verification	+/- 15% of the initial calibration response factor.	+/- 15% of the initial calibration response factor.	Every 24 hr. or at the beginning of an analytical sequence, which ever is more frequent, or as necessary.	Determine cause of ICV failure and correct. Reanalyze ICV and if out of control a new calibration curve must be analyzed.	Target form and raw data.
Continuing Calibration Verification	+/- 15% of the initial calibration response factor. Analyze singly every 10 injections and after the final sample.	+/- 15% of the initial calibration response factor.	Every 10 injections and at the end of an analytical sequence.	Determine cause of failure and correct. Reanalyze calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit.	Target form and raw data.
Instrument Blank	Analyze after analysis of a sample with analyte levels which exceed the upper calibration level.	Analyze after samples when high levels of matrix are suspected.	As necessary.	Reanalyze any samples with suspected carryover.	Raw data
Method Blank	All analytes must be < MDL	All analytes must be less than the Reporting Limit	One method blank per 20 samples or each extraction batch, whichever is more frequent.	Reanalyze blank. If still out of control, re-extract the entire batch of samples unless the detected analyte(s) are not present in the associated samples.	Method Blank Summary and raw data. Narrative comment when necessary.

Method 8330 Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Surrogate Recovery	Minimum of 1 surrogate. Recoveries must fall within the labs established windows.	See latest version of Laucks Testing Labs control limits catalogue. All surrogate recoveries must fall within the generated limits. If laboratory limits are not established due to the implementation of a new surrogate, the default limits of 70% - 130% are used until enough data points are collected to generate new limits.	All samples, method blanks, and QC samples.	Re-extract if surrogate recovery is < the lower control limit. If a poor injection is suspected reanalyze the sample.	Target Surrogate Summary form or special test results. Narrative comment when necessary.
MS/MSD	One per 20 samples or extraction batch which ever is more frequent. Must be per matrix.	See latest version of Laucks control limits located in the QC database.	1 MS/MSD pair per 20 samples or every extraction batch which ever is more frequent.	Per SW 846, if analyte recoveries are out of control in the MS/MSD but are in control in the associated blank spike, no further action is required.	MS/MSD report and raw data. Narrative comment when necessary.
Blank Spike	N/A	See latest version of Laucks Testing Labs control limits in the QC database. All recoveries must fall within the generated limits.	1 per 20 samples or every extraction batch which ever is more frequent.	Re-extract if analyte recoveries are outside of control limits.	Blank Spike report and raw data. Narrative comment when necessary.

APPENDIX V

Elution Order of Target Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
HMX	1	10
RDX	2	8
1,3,5-TNB*	3	1
1,3-DNB	5	2
Tetryl	6	9
NB*	7	1
2,4,6-TNT*	8	4
4-Am-DNT	9	6
2-Am-DNT	10	7
2,4-DNT*	11	4
2,6-DNT*	12	4
2-NT*	13	3
3-NT*	14	3
4-NT*	15	3
Surrogate		
1,2-Dinitrobenzene	4	5

Elution Order of 6 Additional Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (C8+CN) Column
TNX	1	1
2,6-DA-4-NT	2	2
2,4-DA-6-NT	3	3
MNX	4	4
3,5-DNA	6	6
4,4'-TN-AZOXY	7	7
Surrogate		
1,2-Dinitrobenzene	5	5

Elution Order of PETN/NG

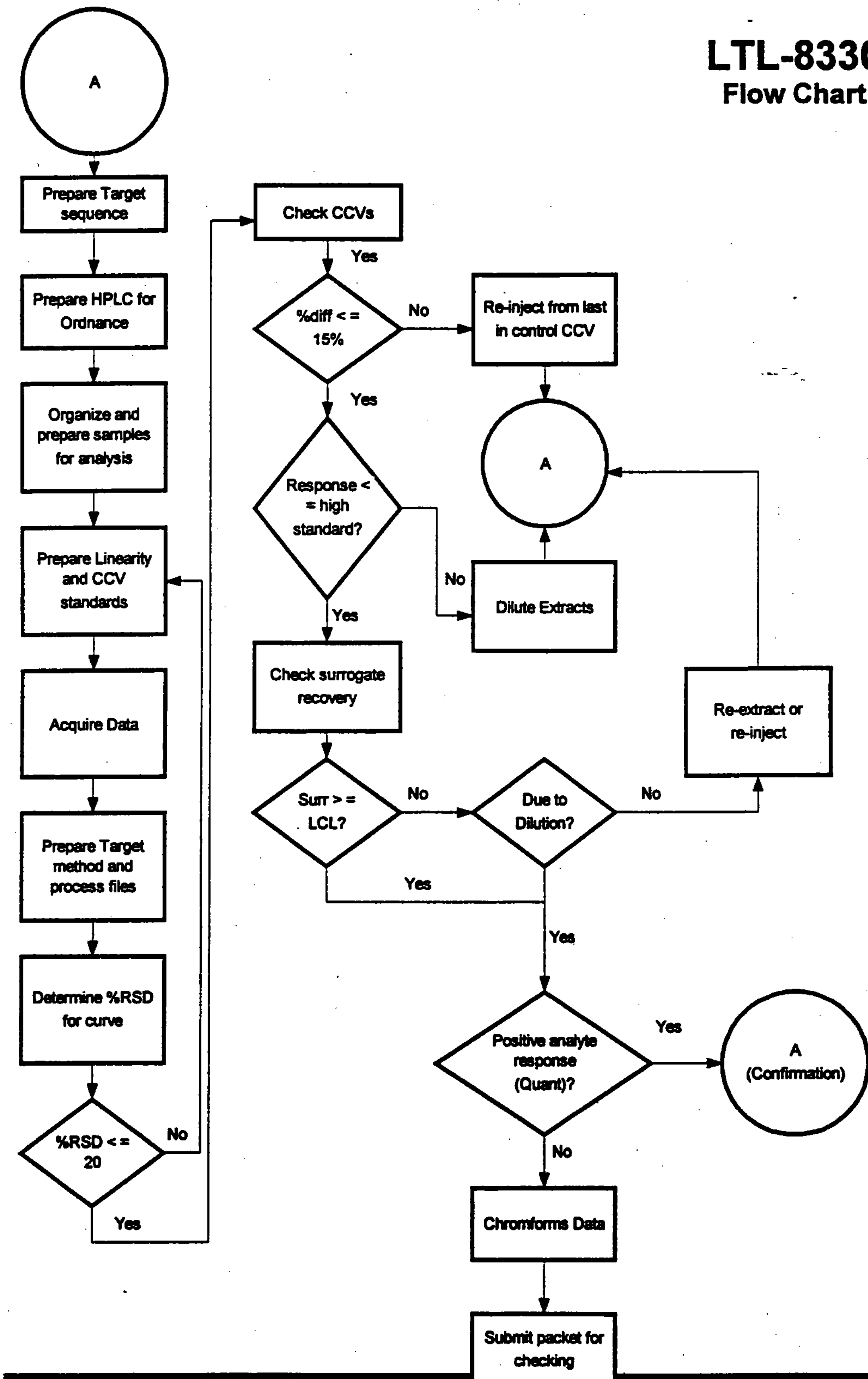
Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
NG	2	2
PETN	3	3
Surrogate		
1,2-Dinitrobenzene	1	1

*Indicates the compound co-elutes on the confirmation (CN) column.

APPENDIX VI

Method 8330 Flow Chart

LTL-8330
Flow Chart



APPENDIX D

HEALTH AND SAFTEY PLAN

APPENDIX E

DATA MANAGEMENT PLAN

1.0 PROJECT PLANNING

A large amount of environmental and physical data has been collected in support of the Installation Restoration (IR) program. TtNUS has the responsibility of managing this data in a basewide relational database and GIS. The contents of the database shall be outlined in the Sitewide Data Catalog (which at a minimum, contains the data fields identified in Attachment 1). The Data Catalog shall outline what data is contained within the database (by investigation, media, etc.), the generator of the data (TtNUS, Corps of Engineers, etc.), and the level of quality of the data where applicable. It should be noted whether or not the analytical data were validated and to what level. It is the responsibility of the TtNUS data manager to coordinate with the NSWC Crane project team in order to keep the Data Catalog current and make available the most recent version to all team members. A copy of the Data Catalog shall be maintained in the project central file at the office of TtNUS. It is the responsibility of the all team members to ensure that the Data Catalog is correct and current and shall notify the TtNUS data manager of any newly generated data that will support the needs of the project.

Prior to every data collection event, the TOM shall call a kick-off meeting to outline the data needs of the task order and to review the data flow process (Attachment 2). Attendees to the kick-off meeting should include the TOM, the Human Health Risk Assessment (HHRA) lead, the Field Operations Leader (FOL), the project chemist, the data management lead and the Geographic Information System (GIS) lead. The data management lead shall distribute a copy of the database checklist (Attachment 3) and shall lead the project team through its contents. The database checklist will allow the project team to determine how the data will be managed and manipulated in order to achieve the project needs and objectives. A completed copy of the database checklist shall be maintained in the project central file and distributed to all members of the project team within seven days of the kick-off meeting.

2.0 NEWLY GENERATED DATA

Upon directive from SOUTHDIV to collect additional site data, the TOM shall coordinate with the designated data management lead and GIS lead for the project. It is the responsibility of the FOL to comply with the sample and location nomenclature outlined in the Work Plan. It is also the responsibility of the FOL to coordinate with the GIS lead to ensure that all survey technical specifications require the proper coordinate system, which is Indiana State Planar - North American Datum 1983 for the horizontal coordinates and National Geodetic Vertical Datum 1988 for the vertical coordinates.

Prior to field mobilization, the FOL shall coordinate with the Sample Management Coordinator (SMC) to initiate a sample tracking process. It is the responsibility of the TOM to ensure that a sampling tracking procedure is implemented. Sample Tracking Request Forms, a sample tracking database example, and example jar labels are included as Attachments 4, 5 and 6, respectively. In the event that a field change has taken place, the FOL is required to complete the Field Task Modification Request (FTMR) that will be forwarded to all members of the project team.

According to all laboratory technical specifications for NSWC Crane, the analytical laboratories will be contractually required to deliver the analytical data in NSWC Crane standard Electronic Data Deliverable (EDD) format (Attachment 7). Particular attention should be paid to the EDD requirements for validated vs. non-valididated data. Once all samples and analyses have been accounted for, the SMC shall forward the analytical data to TtNUS for incorporation into the NSWC Crane database which is located on the Local Area Network (LAN) in Pittsburgh, PA. The NSWC Crane database structure is presented in Attachment 8.

3.0 HISTORICAL DATA

In the event that the NSWC Crane project team decides that existing hardcopy data not outlined in the Data Catalog (Attachment 1) needs to be incorporated into the project database, SOUTHDIV shall provide directive to the appropriate consultant to incorporate the data into the project database. The data management lead shall review the hardcopy data and prepare a summary of the samples and analyses that need to be entered. The format of the summary table should be similar to the sample tracking database provided in Attachment 5. It is the responsibility of the TOM to review the sample summary table and verify that the entry of this data will satisfy the project requirements. The data management lead shall physically edit the hardcopy analytical data to clearly designate which information on the hardcopy needs to be entered into the database. Copies of the marked-up data must be distributed to two separate parties for entry into an Excel spreadsheet. Upon completion of the dual-key entry, the data management lead shall electronically compare the two data files to identify discrepancies and correct the data appropriately. The database should then be queried against the sample summary table to ensure that all pertinent data has been entered and checked for accuracy.

The data management lead shall coordinate with the GIS lead to acquire the sample location data (Attachment 8) for those samples that need to be entered. Sample location maps should be used to digitize the sample locations using the base mapping layer in the GIS. To the extent possible, the GIS lead shall capture, as metadata, the accuracy of the sample location maps used to digitize the location coordinates. If no sample location maps or other positional information exist for the historical data, the project team should evaluate the utility of this data in the NSWC Crane database.

4.0 MAPPING AND GRAPHICS

CADD mapping is generally provided by the activity. We currently do not use metadata to track changes to the mapping. In addition, Tri-Service Spatial Data Standards (TSSDS) are not utilized unless the mapping from the base already incorporates them. TSSDS is not used in the final GIS, based on the view that limited utility is gained from the substantial time required to incorporate the standards.

In addition to CADD mapping, Digital Ortho Quarter (DOQ) Quads, Aerial Photography, and USGS 7.5 minute Quads are obtained. The Quads are obtained from either the USGS or other suppliers, while the aerial photography is provided by the activity. As necessary, the images are warped to the predetermined coordinate system using Microstation. Again, metadata are not used to track the changes. From survey data, sampling locations are organized, and then a sample-vs-location table is built so that the data can be loaded into the sample_data.dbf table (Attachment 8).

5.0 THE ENVIRONMENTAL GEOGRAPHIC INFORMATION SYSTEM (EGIS)

All environmental data collected in support of the NSWC Crane project shall be incorporated into the GIS. The themes, layers and database information contained in the GIS is outlined in the Data Catalog (Attachment 1). The NSWC Crane GIS shall be made available to all members of the project team. CD-ROM EGIS deliverables shall be made available upon request from SOUTHDIV.

6.0 ASSIMILATION OF DATA FROM OUTSIDE SOURCES

When environmental data is collected by a contractor other than TtNUS, it is the responsibility of the SOUTHDIV Remedial Project Manager (RPM) to notify the TtNUS TOM. The RPM should forward a scope of work directing TtNUS to coordinate with the contractor and incorporate their data into the basewide GIS. To the extent possible, the RPM should direct the Navy Contractor to supply the data to TtNUS in the format outlined in Attachment 8. Once TtNUS has incorporated the data into the GIS, a hardcopy report shall be sent to the contractor for verification that all pertinent data have been incorporated in a complete and accurate fashion.

7.0 SOFTWARE

TtNUS will standardize on the following software packages when managing and manipulating data for the NSWC Crane project:

- Data Management - Microsoft Visual FoxPro 6.0
- GIS - ArcView 3.1 (see Attachment 9 for instructions)
- Geostatistics (2-D Kriging) - Geosoft 3.1b
- 3-D Visualization - EVS Pro 3.0
- Ground Water Modeling - GMS
- Statistical Analysis - Statistica 5.1
- Terrain Analysis - TerraModel 9.4.1

8.0 STORAGE OF DATA

TtNUS utilizes Microsoft NT for Networks as its Information Management System (IMS). The NT IMS has a storage capacity of 2 Gigabytes and currently serves over 110 desktop computers. The NT IMS automatically backs-up the system on a daily basis, thereby disallowing more than one day of work being lost should the network crash or malfunction. The database management and GIS groups have been allocated distinct drives on the Local Area Network (LAN). All environmental data for the NSWC Crane Project shall be stored on \\nusrpitbdc1\sdiv\NSWC_Crane subfolder of this drive on the NT Server. All tables, queries, programs and reports shall be saved in the NSWC_Crane.pjx file in Microsoft Visual FoxPro. The NSWC Crane EGIS shall be stored on \\nusrpitbdc1\gis\NSWC_Crane on the NT Server. All ArcView project files (*.apr) shall be documented in a text file called readme_project.txt. This text file shall also be stored on \\nusrpitbdc1\gis\NSWC_Crane.

ATTACHMENT E1

DATA CATALOG

(Minimum Requirements)

DATA CATALOG DATA FIELDS

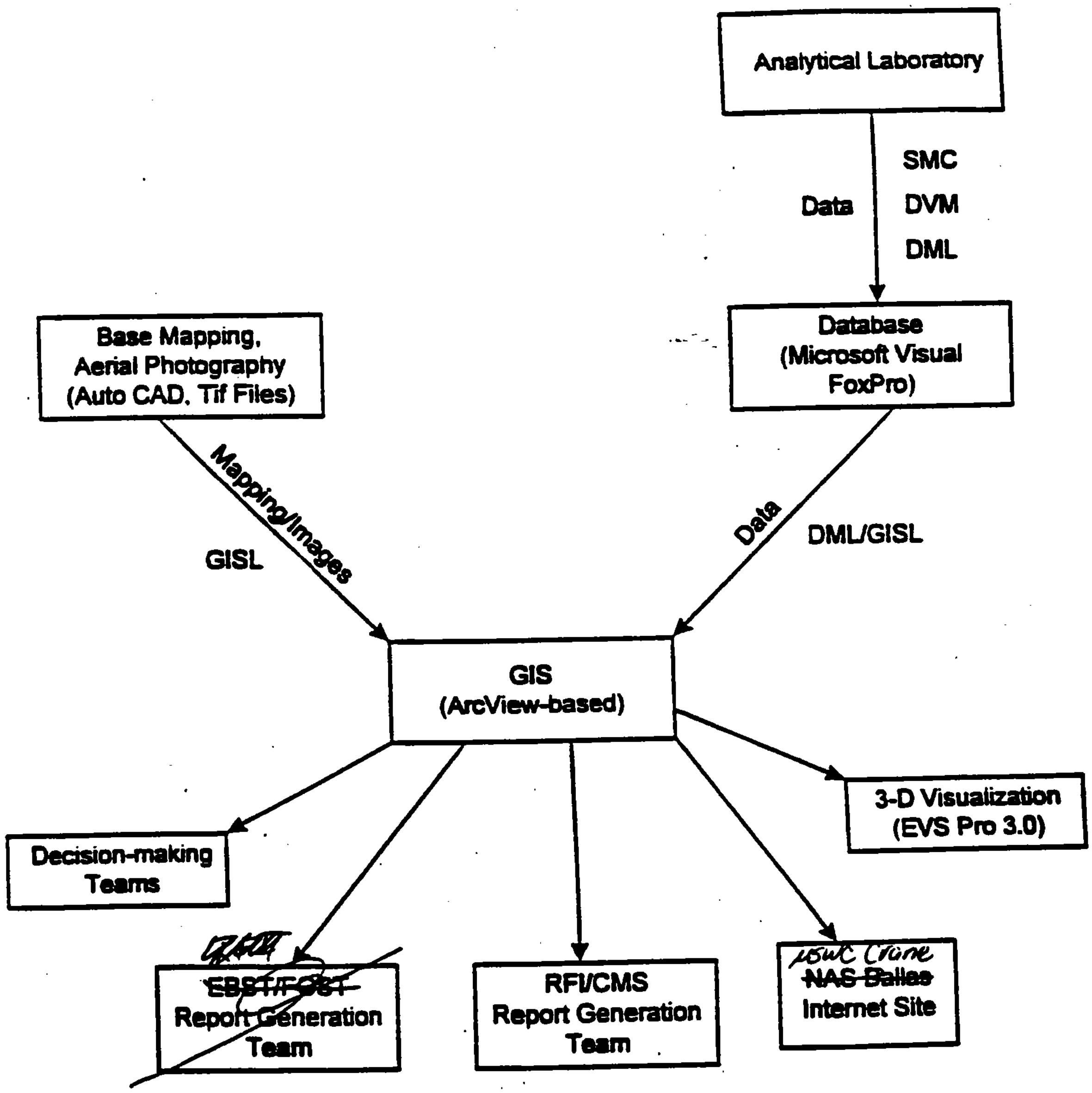
NSWC CRANE
CRANE, INDIANA

Category	RFI Phase	Medium Sampled	Sample Type	No. of Samples	Fraction Analyzed

ATTACHMENT E2

THE DATA FLOW PROCESS

THE DATA FLOW PROCESS



Notes:

SMC = Sample Management Coordinator
DVM = Data Validation Manager
DML = Data Management Leader
GISL - GIS Leader

ATTACHMENT E3

DATABASE CHECKLIST

DATABASE PLANNING CHECKLIST

PROJECT NAME ________________ PROJECT NUMBER ________________

PROJECT MANAGER ________________ PLANNING DATE ________________

1 Provide a general description of the project (regulatory authority, media to be samples, approximate number of samples by media, analyses by media, data evaluation tasks required):

____

____

____

____

____

2. Provide a general description of the sample nomenclature that will be used for samples collected by Brown & Root Environmental:

____

____

____

____

3. Will historical data be entered in the database? Yes No

4 Will historical data be used to define the nature and extent of contamination? Yes No

5. Will historical data be used for risk assessment purposes? Yes No

6. How much historical data exists (i.e., number of samples by matrix, analysis by matrix)?

____

____

____

____

7. In what format will the historical data be provided? Hardcopy Electronic

8. If historical data are in electronic form, what software was used and what is the format?

____

____

____

____

9. If historical data are in hardcopy form, will Form I's, summary tables, or reports be provided? Copies of historical data will be necessary to generate a budget estimate.

____

____

____

____

10. Will Quality Assurance review of historical data be necessary? Yes No

11. If Quality Assurance review of historical data is necessary, describe the scope of the Quality Assurance review:

____

____

____

____

12. Will a GIS database be necessary for the project? Yes No

13. What nomenclature has been (will be) used to identify field duplicate samples?

____

____

____

____

14. Will field duplicate results be averaged and presented as one result in the data base? Will they be presented as distinct results, or will both the average and the distinct results be presented?

____

____

____

____

15. How will the average value for duplicate samples be determined on a matrix-specific basis?

____

____

____

____

16. Are any unvalidated data to be included in the database? Yes No

17. Will unvalidated data be used for defining the nature and extent of contamination? Yes No

18. Will unvalidated data be used for risk assessment purposes? Yes No

19. Are any field screening data to be included in the database? Yes No

20. Will field screening data be used for defining the nature and extent of contamination? Yes No

21. Will field screening data be used for risk assessment purposes? Yes No

22. Will statistical correlation of laboratory and field screening data be necessary? Yes No

23. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to define nature and extent? Yes No

24. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to support the risk assessment? Yes No

25. Will field parameters be included in the database (e.g., pH, conductance, temperature)? Yes No

26. Will statistical correlations be necessary for TCLP versus RAS/SAS data? Yes No

27. Will statistical correlations be necessary for filtered versus unfiltered samples? Yes No

28. Will any other statistical correlations be necessary? Yes No

29. Are there wells that have been screened in different aquifers? Yes No

30. Will data for various aquifers be segregated by depth? Yes No

31. Can the sample nomenclature system be used to identify wells in different aquifers? Yes No

32. Will samples from other matrices (soil, sediment, or surface water) be segregated by depth? Yes No

33. Can the sample nomenclature system be used to identify depth-specificity? Yes No

34. Have any removal actions be performed at the site? Yes No

If removal actions have been performed, plan and cross-sectional views reflecting the extent of the removal action s be provided.

35. Will any composite sample results be included in the database? Yes No

36. If composite samples are included how will they be used for the nature and extent of contamination?

37. If composite samples are included how will they be used for the risk assessment?

38. Will the site be segregated into Areas of Concern, Solid Waste Management Units, etc? Yes No

39. Is the sample nomenclature adequate for such segregation? Yes No

If the sample nomenclature is inadequate for assigning samples to an AOC or SWMU, the Project Manager or designee must provide a base map of tabular summary clearly delineating the relationship between each sample and each AOC/SWMU.

40. Were any temporal samples collected (e.g., quarterly sampling of wells)? Yes No

41. If temporal samples were collected, how will they be used to define the nature and extent of contamination?

42. If temporal samples were collected, how will they be used to support the risk assessment?

43. Are State, Federal, or Regional criteria to be included in data summary tables? Yes No

44. Identify the criteria that must be presented in the summary tables.

45. Will State, Federal, or Regional criteria be used to select COPCs? Yes No

46. Identify the criteria to be used as COPC selection tools.

47. Are filtered and unfiltered surface water samples differentiated? Yes No

48. If such samples are differentiated, how?

49. Which of these samples will be used for the human health risk assessment?

Surface Water Filtered Unfiltered
Groundwater Filtered Unfiltered

50. Which of these samples will be used for the ecological assessment?

Surface Water	Filtered	Unfiltered
Groundwater	Filtered	Unfiltered

51 Will background data be included in the database? Yes No

52. How are background samples identified?

____

____

____

53. Will background results be used to support selection of COPCs? Yes No

53. What statistical analyses will be required for the background data?

____

____

____

54. Will background data be segregated by depth? Yes No

55. What background matrices must be segregated by depth?

____

____

____

56. What format will be used for data presentation (e.g., appendices and summary tables, comprehensive text tables, tag maps, isoconcentration contours, etc.)?

____

____

____

ATTACHMENT E4

SAMPLE TRACKING REQUEST FORM

Sample Tracking and Data Management at Project Inception

PROJECT START-UP CHECKLIST

ATTACHED IS A PROJECT START-UP CHECKLIST (CAN BE FOUND IN DATA MANAGEMENT ROOM). WHENEVER A NEW PROJECT IS STARTED THE TOP PART SHOULD BE FILLED IN. A COPY SHOULD BE RETURNED TO THE DATA MANAGEMENT ROOM. KEEP ORIGINAL FOR YOUR RECORDS TO KEEP TRACK OF WHAT HAS BEEN PROVIDED. IMSG WILL CHECK OFF WHEN ALL INFORMATION IS RECEIVED

FOLLOWING THIS PROCESS WILL IMPROVE THE FOLLOWING:

- TURN-AROUND TIME FOR DELIVERABLES NEEDED WHEN ALL RESULTS HAVE BEEN RECEIVED.
- CONFIDENCE THAT ALL SAMPLE RESULTS HAVE BEEN RECEIVED
- CONSISTENCY OF SAMPLE NOMENCLATURE
- CORRECTNESS OF SAMPLE ATTRIBUTES
- REVIEW OF INVOICES
- ENABLE IMSG PERSONNEL TO BETTER TRACK UPCOMING WORKLOAD

PROJECT START-UP CHECKLIST
INFORMATION NEEDED TO CREATE NEW DATABASE

PROJECT NAME:______________________________

CTO #:________ JOB #:______________

PROJECT MANAGER/CONTACT:______________________

LABELS: Y / N DUE DATE:__________

VALIDATE: Y / N / L DUE DATE:__________

COMBINE WITH HISTORICAL DATA: Y/N

SAMPLE DATA CHECKLIST:

_____ SAMPLE NUMBERS AND ANALYSES (LOCATIONS,DEPTHS)
_____ SECTION OF WORKPLAN PERTAINING TO SAMPLE NOMENCLATURE
_____ LABORATORY/BOTTLE REQUIREMENTS
_____ LAB SPECS
_____ COC'S
_____ SAMPLE LOG SHEETS
_____ DUPLICATE ID'S / ORIGINALS
_____ SURVEY DATA / SAMPLE LOCATION MAPS
_____ BREAKDOWN OF PROJECT BY SITE / MATRIX FOR FUTURE PRINTOUTS
_____ TABLE HEADERS (SEE EXAMPLE)

TO BE COMPLETED BY IMSG:

_____ FINAL RESULTS GIVEN TO _______________ (PM/IMSG)
DATE:____________
_____ SAMPLE DATA LOADED INTO NEW/EXISTING PROJECT DATABASE
_____ RESULTS LOADED INTO NEW/EXISTING PROJECT DATABASE
PATHNAME OF PROJECT DATABASE:_______________
_____ DATA LOADED INTO GIS

ATTACHMENT E5

SAMPLE TRACKING DATABASE EXAMPLE

CTO 020 SDG U06972

07/15/98

Proj Name	Job No	Sdg	Sample Number	Lab Id	Fraction	Sort	Lab Rec	B&R Rec	Turn-Time	WO No	Laboratory
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	LV	LV	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	MISC	CN	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	MISC	CR6	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	OS	OS	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PAH	PAH	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PEST/PCB	PCB	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PEST/PCB	PEST	06/03/98	07/14/98	41	9806G972	RECRA

ATTACHMENT E6

EXAMPLE SAMPLE JAR LABELS

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: TCL Volatiles
Sampled by: | Laboratory: RECRA

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: TCL Semivolatiles, TAL Metals, Cyanide
Sampled by: | Laboratory: RECRA

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: OTTO Fuel
Sampled by: | Laboratory: GEL

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTC
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLEST
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLES
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLE
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLE
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

ATTACHMENT E7

ELECTRONIC DATA DELIVERABLE REQUIREMENTS FOR ANALYTICAL LABORATORIES

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to provide 3.5" high density diskette(s) containing separate database (DBF) file the format specified in this Attachment. The electronic deliverable includes all environme samples, sample dilutions, sample reanalyses, and laboratory quality control samples. **All entrie the electronic deliverable must agree exactly with the final entries reported on the hardc data package sample result summaries.** Any corrections made to the hardcopy data must als made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must als designated; laboratory QC non-compliance codes are not to be depicted.

Each diskette is to be properly labeled with the laboratory name, project name, file name(s), laboratory point of contact. Electronic files should be delivered in the same fashion as are the copy data packages. A separate .dbf file shall be made for each analytical fraction (by method) each sample delivery group (SDG). The files shall be named with the first character being analytical fraction designator, followed by an underscore, followed by the SDG name. For exa the file for the volatile fraction for SDG BR001 should be named V_BR001.DBF. Additionally laboratory must provide a hardcopy listing all electronic files saved to the diskette, indicating analytical fraction and matrix the file data contained therein pertain to. All electronic deliverables are due within the same time established for the associated hardcopy data package

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Re Form displayed on the next page of this Attachment. Electronic deliverables are not considered complete without the accompanying Quality Assurance Review Form.

I ______________________________, as the designated Quality Assurance Officer, hereby attest t all electronic deliverables have been thoroughly reviewed and are in agreement with the associa hardcopy data. The enclosed electronic files have been reviewed for accuracy (including signific figures), completeness and format. The laboratory will be responsible for any labor time necessar correct enclosed electronic deliverables that have been found to be in error. I can be reached at (______)______________if there are any questions or problems with the enclosed electr deliverables.

Signature:______________________________ Title: ______________________________ Date: ______

Revision 4
KAC 11-11-97

The analytical data shall be delivered electronically in a Dbase III file format (filename.dbf). T exact structure of the database is described in the table below. It shall be the responsibility of laboratory to ensure that all electronic entries are in strict accordance with the information provic on the Form I.

An example database shall be sent for review prior to the first electronic deliverable in Dbase format. The example file will be examined for completeness and comments will be sent to laboratory. Any questions regarding the electronic deliverable shall be directed to Patrick Hoope Brown and Root Environmental (412)921-8250.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
SAMPLE_NO	C	25	Field sample ID as listed on the chain-of-custody. The sample number indicated in this field should never be truncated. The only exception for this field not matching the chain-of-custody is for reana and matrix spike results in which a RE or MS suffix will be added to sample number respectively.
TRUNCATE	C	15	If the field sample ID listed on the Chain of Custody is truncated by the laboratory for use with the laboratory software. the truncated sample ID should appear in this field.
LAB_ID	C	15	Laboratory number for the given sample.
LABORATORY	C	25	Laboratory name.
BATCH_NO	C	10	Laboratory code for batch of samples included in a given run.
ASSOC_BLNK	C	15	Laboratory name of the method blank associated with that particula batch of samples.
QC_TYPE	C	15	Normal Environmental Sample = "NORMAL". Laboratory Duplicat "DUPLICATE". Matrix Spike = "MS". Matrix Spike Duplicate = "MS Laboratory Control Sample = "LCS". Laboratory Control Sample Duplicate = "LCSD". Method Blank = "M_BLANK". Preparation Bl "P_BLANK".
SAMP_DATE	D	8	Date of sample collection as indicated on the Chain of Custody. Example: 11/07/93.
REC_DATE	D	8	Date sample was received by the laboratory.
EXTR_DATE	D	8	Date sample was extracted or prepared by the laboratory.
ANAL_DATE	D	8	Date sample was analyzed by the laboratory.
RUN_NUMBER	N	2 (0)	The number of the analytical run for a given sample in sequence. example. if a sample is diluted and reanalyzed. the original run nu would be 1 and the reanalysis would be 2.
SDG	C	15	Sample delivery group identifier assigned by the laboratory. This number should <u>exactly</u> match the SDG designated on the hardcop package.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
PROJECT_NO	C	10	Identification of Project Number or CLEAN Task Order (CTO) num
PROJ_MNGR	C	25	The Brown & Root Project Manager's last name. followed by a comm followed by the first initial of the Project Manager (e.g. Hutson. D).
PARAMETER	C	45	Chemical or analyte name exactly as reported on Form I.
CAS_NO	C	10	Chemical Abstract Service number for the parameter listed. The C number should be reported exactly as it is listed in publications suc the Merck Index. This field should be left blank for those paramete having CAS numbers (e.g. Total Organic Carbon).
FRACTION	C	5	Metals = 'M', Volatiles = 'OV', Semivolatiles/BNAs = 'OS', Pesticides 'PEST', Herbicides = 'HERB', Polychlorinated Biphenyls = 'PCB', Explosives = 'EXP', Any petroleum hydrocarbon or fuel = 'TPH', We Chemistry = 'WET', Radionuclide = 'RAD', Miscellaneous = 'MISC'
METHOD	C	20	Analytical method used to quantitate parameter concentrations as in the laboratory technical specification (e.g. '8270A' for SW-846 M 8270A.
LAB_RESULT	N	20 (6)	Reported value in units specified in the UNITS field containing the proper number of significant digits. The % Recovery shall be place this field for matrix spike and laboratory control sample results.
UNITS	C	5	The units of measure as reported on the Form I.
LAB_QUAL	C	2	The laboratory qualifier as reported on the Form I. For example, a qualifier should be used for all nondetected results.
IDL	N	15 (6)	Instrument detection limit in units specified in the UNITS
MDL	N	15 (6)	Method detection limit in units specified in the UNITS field and method specified in the METHOD field.
CRDL_CRQL	N	15 (6)	Contract Required Detection/Quantitation Limit in the units speci the UNITS field. RDL for non-CLP parameters.
DIL_FACTOR	N	6 (1)	Dilution factor.
PCT_MOIST	N	5 (1)	Percent moisture for soil samples: blank for water samples.
COMMENTS	C	20	Analytical result qualifier or comment other than that listed in th LAB_QUAL field. Example: 'Reanalysis'.

C = Character string (everything shall be reported in capital letters)
N = Numeric string (decimal places are in parentheses in field width column)
D = Date (Ex: 05/25/97)

ATTACHMENT E8

DATABASE STRUCTURE

Database Structure

The NAS Dallas master database shall contain 18 standard tables to store all chemical. geological. and hydrogeological data. The structure, indexes, primary keys, and relations for each table are defined below.

TABLE: well
PRIMARY KEY: location

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name.
post_id	C (20)	Location name as derived from original source document.
instal_date	D (8)	Date the monitoring well was installed. Null for other location types.
loc_type	C (4)	Type of location.
Northing	N (15,4)	Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	N (15,4)	Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
horiz_datum	C (8)	Datum in which the horizontal coordinates were derived.
grnd_surf	N (15,4)	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
vert_datum	C (25)	Datum in which the vertical coordinates were derived.
datum_state	C (2)	State for which datum was developed
Surveyed	L (1)	Logical field denoting whether positional data were surveyed or digitized.
Surveyor	C (50)	Company who performed the survey.
survey_date	D (8)	Date in which survey was performed.
surv_method	C (25)	Surveying method used.
Longitude	N (15,4)	Longitude
Latitude	N (15,4)	Latitude

Table Indexes

INDEX	TYPE
Location	Primary
Hd	Regular
Vd	Regular
loc_type	Regular

Table Relations:

Relation 1
*RelatedChild loc_type
*RelatedTable loc_type_vvl
*RelatedTag loc_type

Relation 2
*RelatedChild hd
*RelatedTable horiz_datum_vvl
*RelatedTag hd

Relation 3
*RelatedChild vd
*RelatedTable vert_datum_vvl
*RelatedTag vd

TABLE: loc_type_vvl - Valid value list for LOC_TYPE field in the well table.
PRIMARY KEY: loc_type

Table Structure

FIELD	TYPE	DESCRIPTION
loc_type.	C (4)	Location type
Description	C (40)	Description of location type

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: horiz_datum_vvl - Valid value list for HORIZ_DATUM field in the well table.
PRIMARY KEY: hd (horiz_datum)

Table Structure

FIELD	TYPE	DESCRIPTION
Horiz_datum	C (15)	Datum in which x,y coordinates reflect

Table Indexes

INDEX	TYPE
hd (horiz_datum)	Primary

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: vert_datum_vvl - Valid value list for VERT_DATUM field in the well table.
PRIMARY KEY: vd (vert_datum)

Table Structure

FIELD	TYPE	DESCRIPTION
Vert_datum	C (15)	Datum in which z coordinate reflects

Table Indexes

INDEX	TYPE
vd (vert_datum)	Primary

TABLE: sample_data - Sample data table
PRIMARY KEY: nsample

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name.
Matrix	C (4)	Sample matrix
Nsample	C (35)	Unique sample identification
Sample	C (25)	Sample identification as designated on Chain-of-Custody
Sacode	C (8)	Sample code for reference to field duplicates
top_depth	N (5,1)	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
Bottom_depth	N (5,1)	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. *Rule Expression*: iif(bottom_depth>0.top_depth<=bottom_
qc_type	C (2)	Quality control type

Status	C (10)	Status of sample location – Normal or excavated
sample_date	D (8)	Date in which sample was collected
Validated	L (1)	Logical field denoting whether or not data validation was performed on sample
coll_method	C (10)	Sample collection method
cto_proj	C (5)	Clean task order (Navy) or project number in which the sample was collected (e.g. "129")
proj_manager	C (25)	Internal projcect manager for which the data was originally generated (e.g. "Hutson, D.").

Table Indexes

INDEX	TYPE
Location	Regular
Nsample	Primary
Sacode	Regular
Matrix	Regular
Status	Regular
qc_type	Regular
coll_meth	Regular

Table Relations:

<u>Relation 1</u>
*RelatedChild sacode
*RelatedTable sacode_vvl
*RelatedTag sacode
<u>Relation 2</u>
*RelatedChild qc_type
*RelatedTable qc_type_vvl
*RelatedTag qc_type
<u>Relation 3</u>
*RelatedChild matrix
*RelatedTable matrix_vvl
*RelatedTag matrix
<u>Relation 4</u>
*RelatedChild location
*RelatedTable well
*RelatedTag location
<u>Relation 5</u>
*RelatedChild coll_meth
*RelatedTable coll_method_vvl
*RelatedTag coll_meth

<u>TABLE:</u> sacode_vvl - Sample code valid value list for SACODE field in sample_data.dbf
<u>PRIMARY KEY:</u> sacode

Table Structure

FIELD	TYPE	DESCRIPTION
Sacode	C (8)	Sample code designating whether sample is a normal environmental sample, a field duplicate or the average of field duplicate pairs
Description	C (30)	Description of sacode entry

Table Indexes

INDEX	TYPE
Sacode	Primary

TABLE: qc_type_vvl - Quality control valid value list for QC_TYPE field in sample_data.dbf
PRIMARY KEY: qc_type

Table Structure

FIELD	TYPE	DESCRIPTION
qc_type	C (10)	Quality control type
Description	C (30)	Description of quality control type

Table Indexes

INDEX	TYPE
qc_type	Primary

TABLE: matrix_vvl - Matrix valid value list for MATRIX field in sample_data.dbf
PRIMARY KEY: matrix

Table Structure

FIELD	TYPE	DESCRIPTION
Matrix	C (4)	Sample matrix
Description	C (25)	Description of sample matrix code

Table Indexes

INDEX	TYPE
Matrix	Primary

TABLE: well_completion
PRIMARY KEY: None

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name
top_casing	N (8,2)	Elevation of top of well casing in vertical datum found in VERT_DATUM in the well table
hole_diameter	N (5,1)	Diameter of the drilled hole in inches ***Rule Expression:*** hole_diameter>casing_id.AND.hole_diameter>casing_od
scr_aquifer	C (30)	Aquifer name in which the screen resides
screen_material	C (15)	Type of material in which the screen is constructed from
scrn_slot_size	N (5,1)	Screen slot size in thousandths of an inch
scrn_top_depth	N (5,2)	Depth below ground surface to the top of the screen (in feet)
scrn_bot_depth	N(5,2)	Depth below ground surface to the bottom of the screen ***Rule Expression:*** if(scrn_bot_depth>0,scrn_top_depth<scrn_bot_depth)
scrn_top_elev	N (6,2)	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table.
scrn_bot_elev	N (6,2)	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table. ***Rule Expression:*** if(scrn_bot_elev>0.scrn_top_depth>scrn_bot_depth)
drill_method	C (15)	Drilling method for well installation
Contractor	C (20)	Drilling contractor
casing_material	C (15)	Type of material in which the casing is constructed from
depth_to_seal	N (8,2)	Depth below ground surface to seal (in feet)

seal_material	C (15)	Type of material in which the seal is constructed from
fill_top_depth	N (6.2)	Depth below ground surface to the top of fill material (in fe
fill_bot_depth	N (6.2)	Depth below ground surface to the bottom of fill material (in feet). *Rule Expression*: if(fill_bot_depth>0.fill_top_depth<scrn_bot_depth)
fill_material	C (20)	Type of material used for fill.
comments	M (4)	Geologist's comments

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:

Relation 1

*RelatedChild location
*RelatedTable well
*RelatedTag location

TABLE: lithology
PRIMARY KEY: None

FIELD	TYPE	DESCRIPTION
location	C (25)	Unique location name
top_lithology	N (6,2)	Depth in feet below ground surface to the top of lithologic unit
bottom_lithology	N (6,2)	Depth in feet below ground surface to the bottom of litholo... unit
uscs_code	C (5)	Unified Soil Classification Service Code for lithology type
blow_counts	C (8)	Number of blow counts recorded on boring log
description	C (80)	Geologist's description of lithology
comments	M (4)	Geologist's comments.

Table Indexes

INDEX	TYPE
location	Regular
uscs_code	Regular

Table Relations:

Relation 1

*RelatedChild location
*RelatedTable well
*RelatedTag location

Relation 2

*RelatedChild uscs_code
*RelatedTable lithology_vvl
*RelatedTag uscs_code

TABLE: lithology_vvl - Lithology valid value list for USCS_CODE field in lithology.dbf
PRIMARY KEY: uscs_code

Table Structure

FIELD	TYPE	DESCRIPTION
uscs_code	C (4)	Unified Soil Classification Service Code for lithology type
descript	C (70)	Description of lithology for given USCS code

Table Indexes

INDEX	TYPE
uscs_code	Primary

TABLE: coll_method_vvl - Collection method valid value list for COLL_METHOD field in sample_data.dbf
PRIMARY KEY: coll_meth

Table Structure

FIELD	TYPE	DESCRIPTION
coll_method	C (10)	Sample collection method

Table Indexes

INDEX	TYPE
coll_method	Primary

TABLE: cas_vvl - CAS number valid value list for CAS field in analytical results.dbf
PRIMARY KEY: cas

Table Structure

FIELD	TYPE	DESCRIPTION
parameter	C (40)	Parameter or chemical name
cas	C (11)	Chemical Abstracts Service Number

Table Indexes

INDEX	TYPE
parameter	Regular
cas	Primary

TABLE: analytic_results
PRIMARY KEY: nfp (nsample+fraction+parameter)

Table Structure

FIELD	TYPE	DESCRIPTION
nsample	C (35)	Unique sample identification
lab_id	C (15)	Laboratory sample identification
laboratory	C (25)	Laboratory name
batch_no	C (10)	Analytical batch number
assoc_blnk	C (15)	Associated blank
extr_date	D (8)	Extraction date
anal_date	D (8)	Analysis date
run_number	I (4)	Sequential analytical run number
sdg	C (15)	Sample delivery group
parameter	C (45)	Parameter or chemical name (using IUPAC nomenclature where appropriate)
cas	C (11)	Chemical Abstracts Service Number
fraction	C (5)	Analtytical fraction
method	C (20)	Analytical method
lab_result	N (20.6)	Analytical result as reported by the laboratory
lab_qual	C (5)	Qualifier as reported by the laboratory
val_res	N (20.6)	Final result (via validation or otherwise)

result	C (20)	Final analytical result with the correct number of significant figures
val_qual	C (3)	Validation qualifer (null if data were not validated)
qual	C (3)	Final qualifer (validation or otherwise)
units	C (5)	Units of measure for the RESULT field
idl	N (15,6)	Instrument detection limit (same units as UNITS field)
mdl	N (15,6)	Method detection limit (same units as UNITS field)
crdl_crql	N (15,6)	Contract required detection/quantitation limit (same units as UNITS field)
dil_factor	N (6,1)	Dilution factor
pct_moist	N (5,1)	Percent moisture
comments	C (20)	Comments from laboratory analyst

Table Indexes

INDEX	TYPE
nfp	Primary
units	Regular
qual	Regular
fraction	Regular
parameter	Regular
nsample	Regular
cas	Regular

Table Relations:

<u>Relation 1</u>
*RelatedChild cas
*RelatedTable cas_vvl
*RelatedTag cas
<u>Relation 2</u>
*RelatedChild units
*RelatedTable units_vvl
*RelatedTag units
<u>Relation 3</u>
*RelatedChild qual
*RelatedTable qual_vvl
*RelatedTag qual
<u>Relation 4</u>
*RelatedChild fraction
*RelatedTable fraction_vvl
*RelatedTag fraction
<u>Relation 5</u>
*RelatedChild parameter
*RelatedTable para_vvl
*RelatedTag para
<u>Relation 6</u>
*RelatedChild nsample
*RelatedTable sample_data
*RelatedTag nsample

TABLE: units_vvl - Units valid value list for UNITS field in analytical_results.dbf
PRIMARY KEY: Units

Table Structure

FIELD	TYPE	DESCRIPTION
Units	C (8)	Units of measure for chemical analysis
Description	C (20)	Description of units

Table Indexes

INDEX	TYPE
Units	Primary

TABLE: qual_vvl
PRIMARY KEY: qual - Qualifier valid value list for QUAL field in analtyic_results.dbf

Table Structure

FIELD	TYPE	DESCRIPTION
Qual	C (5)	Final QA qualifier
Description	C (60)	Definition of qualifier

Table Indexes

INDEX	TYPE
Qual	Primary

TABLE: fraction_vvl - Analytical fraction valid value list for FRACTION field in analytic_results.dbf
PRIMARY KEY: fraction

Table Structure

FIELD	TYPE	DESCRIPTION
Fraction	C (10)	Analytical fraction
Description	C (35)	Description of fraction

Table Indexes

INDEX	TYPE
Fraction	Primary

TABLE: para_vvl
PRIMARY KEY: parameter

Table Structure

FIELD	TYPE	DESCRIPTION
Para	C (60)	Parameter or chemical name
frac_name	C (35)	Analytical fraction for given parameter

Table Indexes

INDEX	TYPE
Para	Primary

TABLE: fluid
PRIMARY KEY: None

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name
Fluid_date	D (8)	Date measurement was taken
meas_elev	N (8.2)	Measuring point elevation
dep_to_water	N (6.2)	Depth below ground surface to water table (in feet)
dep_to_fp	N (6.2)	Depth below ground surface to free product (in feet)
elev_water	N (8.2)	Elevation of water level
elev_fp	N (8.2)	Elevation of free product
prod_thick	N (6.2)	Product thickness in feet

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location

ATTACHMENT E9

ARCVIEW GIS STRUCTURE

ArcView GIS Structure

The NAS Dallas ArcView GIS shall have the following directory structure and database table structure.

Part One: Directory Structure

The following table defines the directory structure and major file names/types located within each directory.

Main subdirectory	First tier subdirectories	Second tier subdirectories	Files/Types
p:\gis\project name\	database\		coordinate.dbf cross_reference.dbf res_gw.dbf res_so.dbf res_sd.dbf res_sw.dbf well_completion.dbf
		criteria\	crit_gw.dbf crit_so.dbf crit_sd.dbf crit_sw.dbf crit_des.dbf
	mapping\	aerial\	registered aerial photos
		drg\	USGS Digital Raster Graphic
		image\	GeoStatistic Layers, pictures of sites, equipment, EVS, and all other raster files.
		dwg\	AutoCAD files
		dgn\	Microstation files
		shp\	samp_gw.shp .dbf .shx samp_so.shp .dbf .shx samp_sd.shp .dbf .shx samp_sw.shp .dbf .shx and all other AV shape files
	working\	database\	files used to generate specific drawings will be put under the working subdirectory in subdirectories similar to database & mapping. These will not be included in CD deliverable.
		mapping\	same as above

Part Two: Database Table Structure

The ArcView GIS will contain separate database tables to store analytical, criteria, and coordinate information. The structure of these tables is presented below.

Analyticl Data Table

The following table lists all the fields contained in the analytic database table.

FIELD :	VISIBLE	ALIAS	DESCRIPTION
site	Yes	Site or SWMU	Site or SWMU
location	Yes	Location	Unique location name
nsample	Yes	Sample	Unique sample identification
sample	No		Sample identification as designated on Chain-of-Custody
sample_date	Yes	Sample Date	Date in which sample was collected
matrix	Yes	Matrix	Sample matrix
sacode	Yes	Sample Code	Sample code for reference to field duplicates
depth	Yes	Depth	Depth in feet to the middle of the sample interval. Applicable for soil and sediment samples.
top_depth	Yes	Top Depth	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
bottom_depth	Yes	Bottom Depth	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. Rule Expression: iif(bottom_depth>0,top_depth<=bottom_depth)
parameter	Yes	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate)
cas	Yes	CAS	Chemical Abstracts Service Number
fraction	Yes	Fraction	Analytical fraction
val_res	Yes	Numeric Result	Final result (via validation or otherwise)
qual	Yes	Qualifier	Final qualifier (validation or otherwise)
units	Yes	Units	Units of measure for the RESULT field
method	Yes	Method	Analytical method
status	Yes	Status	Status of sample location – Normal or excavated
validated	Yes	Validated	Logical field denoting whether or not data validation was performed on sample
coll_method	Yes	Collection Method	Sample collection method
cto_proj	Yes	CTO	Clean task order (Navy) or project number in which the sample was collected (e.g. "129")
proj_manager	Yes	Project Manager	Internal project manager for which the data was originally generated (e.g. "Hooper, P.").
lab_id	No	Laboratory ID	Laboratory sample identification
laboratory	No	Laboratory	Laboratory name
batch_no	No	Batch Number	Analytical batch number

assoc_blnk	No	Associated Blank	Associated blank
extr_date	No	Extraction Date	Extraction date
anal_date	No	Analysis date	Analysis date
run_number	No	Run Number	Sequential analytical run number
sdg	No	SDG	Sample delivery group
lab_result	No	Result	Analytical result as reported by the laboratory
lab_qual	No	Lab Qualifier	Qualifier as reported by the laboratory
result	No	String Result	Final analytical result with the correct number of significant figures
val_qual	No	Validation Qualifier	Validation qualifier (null if data were not validated)
idl	No	Detection Limit	Instrument detection limit (same units as UNITS field)
mdl	No	Detection Units	Method detection limit (same units as UNITS field)
crdl_crql	No		Contract required detection/quantitation limit (same units as UNITS field)
dil_factor	No	Dilution factor	Dilution factor
pct_moist	No	Percent moisture	Percent moisture
ourresult	No		
qc_type	No		Quality control type
comments	No	Comments	Comments from laboratory analyst

Criteria Table

Each medium will have a criteria table to specify the applicable criteria for all parameters.

FIELD	ALIAS	DESCRIPTION
parameter	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate)
epa_mcl	None	Federal MCL – groundwater

Note: usually there will be many criteria fields. This example table only shows the "epa_mcl" criteria field.

Criteria Description Table

This table stores the definition or description of all standards and criteria used in the project. For example, epa_mcl's media would be GW, description would be "Federal Maximum Contaminant Level".

FIELD	Visible	DESCRIPTION
Field	Yes	
Media	Yes	
Descript	Yes	

Coordinate Table

The coordinate table holds all the geographic position information of sampling locations

FIELD	Visible	ALIAS	DESCRIPTION
Location	Yes	None	Unique location name.
post_id	Yes	Location Designation	Location name as derived from original source document.
instal_date	No	Installation Date	Date the monitoring well was installed. Null for other location types.
loc_type	Yes	Location Type	Type of location. Example MW, HP, etc.
northing	Yes		Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	Yes		Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Grnd_surf	Yes	Ground Surface Elevation	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
horiz_datum	Yes	Horizontal Datum	Datum in which the horizontal coordinates were derived.
Vert_datum	Yes	Vertical Datum	Datum in which the vertical coordinates were derived.
Datum_state	Yes	Coordinate System	State for which datum was developed
Surveyed	Yes		Logical field denoting whether positional data were surveyed or digitized.
Surveyor	Yes		Company who performed the survey.
Survey_date	No	Survey Date	Date in which survey was performed.
Surv_method	No	Survey Method	Surveying method used.
Longitude	No		Longitude
Latitude	No		Latitude
gw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
Sd_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
So_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
Sw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
_nullflags	No		Various fields are put in by database starting here and followed by several fields. Make all of these invisible

APPENDIX F

FIELD AUDIT CHECKLIST
(EXAMPLE)

Location: NSWC Crane, Crane, Indiana

Project: NSWC Crane DR Navy/ORR, CTO56

Date of Audit: ____________________

Instructions: Record answers to questions below, providing comments as required to clarify the answers.

QA/QC Procedures

1. Were any field observations, deficiencies, non-conformances, or complaints recorded by the site QA/QC Officer or other personnel?
   If so, summarize below.

   ____
   ____
   ____
   ____
   ____

2. Based on personnel interview, did any variances from the project planning documents occur? If so, what were they?

   ____
   ____
   ____
   ____

3. Were field modification records pertinent to the above initiated in an appropriate manner?

   ____

4. If applicable, were corrective action plans implemented (according to proper procedure)?

   ____
   ____
   ____

5. Were field QC samples obtained with the frequency specified in the QAPP?

   ____

6. For all sites, were field duplicates submitted "blind" to the laboratory?

____
____

7. For all sites, are sufficient replicate aliquots of samples designated to the laboratory for the matrix spike/duplicate analyses specified in the QAPP?

____
____
____

Soil Sampling

8. Are the sampling devices designated in the QAPP or applicable TtNUS SOP being used?

____
____
____
____

9. Was the following information recorded in the boring logs or the field notebook?

For soil classification:

Was the USCS classification and soil type (clay, silt, sand) indicated?

____

Were the following characteristics indicated per the relevant TtNUS SOP CTO 56-3 sections?

color ______________________
soil type ______________________
relative density and consistency ______________________
weight percentage ______________________
moisture ______________________
stratification ______________________
texture/fabric/bedding ______________________

10. For surface soil samples obtained by hand auger or scoop or trowel, were the following practices followed?

area cleared of loose debris prior to sampling ______________________
location marked with numbered stake or pin flag ______________________
sketch approximate locations of sample points in site notebook ______________________

Equipment Decontamination Procedures

11. Has an adequate pre-determined area for steam cleaning of equipment been established?

____

____

12. Is the decontamination (decon) area lined and/or bermed?

____

____

13. Are hand augers decontaminated as required?

____

____

14. Was steam cleaning conducted:

prior to commencement of field activities? ____________________
between boring/pit locations? ____________________
at the end of field activities? ____________________

15. Verify that all sampling equipment not subject to steam cleaning (e.g., trowels, mixing bowls, etc.) are subjected to decontamination per the sequence outlined in the project planning documents.

____

____

Waste Handling Procedures

16. Were cuttings or fluids disposed of in accordance with project planning documents (i.e., discharged to ground, drummed, or tanked)?

____

17. Do the project planning documents provide for the disposal of Personal Protective Equipment (PPE) by double-bagging and discard?

____

18. By what method are PPE disposed of?

____

19. If applicable, were used spill-containment materials containerized or otherwise acceptably disposed of?

____

____

Sample Handling

20. Are the appropriate containers provided by the laboratory being used for each sample?

____

____

____

21. Has the temperature blank been handled properly and one submitted with each cooler of samples?

____

____

22. Have equipment rinsate blanks of the proper type and frequency been obtained?

____

____

23. Have source water blanks been obtained from water sources applicable to the field effort?

____

____

24. Have the rinsate and source water blanks been designated for the same analyses as the associated samples?

____

____

25. Has sample custody been maintained with regard to the following criteria:

A sample is under an individual's custody if:

- it is in the individual's actual possession
- it is in the individual's view after possession
- it was locked up to prevent tampering
- it was placed in a designated and identified secure area

(The sample remains in the individual's custody until it is entrusted to a laboratory courier or commercial express carrier.)

____

____

Documentation

26. Are all sample logs complete (i.e., containing all information stipulated in SOP CTO 56-4)?

____

____

27. Have chain-of-custody (COC) forms been filled out for all samples, including field quality control samples?

____

____

28. Have the COC forms been signed by the appropriate individual at each step that the samples are relinquished?

____

____

29. Have the COC forms been filled-out using black waterproof ink?

____

____

30. If the COC form or other field document was corrected, was a line drawn through the information and was the change dated and initialed? (Use of white-out or erasure is not permitted.)

____

____

31. Have the appropriate analyses (per the project planning documents) been properly designated for each sample on the COC form?

32. Have all sample labels been filled out appropriately and completely?

33. Have sample tags been properly completed and attached securely to samples?

34. Have all sample labels been filled out using indelible ink?

35. Do the sample identifications agree between the sample log, field notebook, sample label and COC form?

36. When applicable, have the name of the photographer, date, time, site location, and site description been entered sequentially into the site logbook as documentative photographs of the sampling have been taken?

37. Has the following information (at minimum) been recorded in the site logbook:

- arrival/departure of site visitors
- arrival/departure of equipment
- sample pickup, COC form numbers, carrier company, time
- sampling activities/sample log sheet numbers.
- start/completion of boreholes, trenches, monitoring wells
- health and safety issues

38. Is the site logbook a bound notebook with consecutively numbered pages that cannot be easily removed?

____

39. As required by SOP CTO 56-4, does the cover of the site logbook contain the following information?

project name	____________
project number	____________
contractor (or Teaming firm) name	____________
sequential book number	____________
start date	____________
end date	____________

40. As required by SOP CTO 56-4, has the following information been recorded at the beginning of each day?

date	____________
start time	____________
weather conditions	____________
all field personnel present	____________
any visitors present	____________

41. Do the site logbook entries summarize the daily activities and refer to other site notebooks or log sheets where applicable?

____

42. Have all site logbook entries been made in black indelible ink?

____

43. If a logbook entry was corrected, was a line drawn through the information and was the change dated and initialed? (Use of white-out or erasure is not permitted.)

____

44. Did the individual making the logbook entry sign it?

____

45. Did the Field Operations Leader sign all logbook pages utilized that day at the end of each day?

____

____

Auditor Name:______________________________

Auditor Signature:______________________________

Date:______________________________

APPENDIX G

ANALYSIS OF VARIANCE (ANOVA)

APPENDIX G PARAMETRIC AND NON-PARAMETRIC ANALYSIS OF VARIANCE

In general, the purpose of analysis of variance (ANOVA) testing is to identify statistically significant differences between data means. If there are significant differences between the data sets, the calculated p level will be less than (1- (level of significance)). For example, at a 5% level of significance (95% confidence level), a p level < 0.05 (or 1 - .95) indicates that there are statistically significant differences between the data sets.

ANOVA testing can take two general forms – parametric and non-parametric. Each is described below.

PARAMETRIC ANOVA

Parametric ANOVA is based on an underlying assumption that the data sets being compared have the same underlying distribution Assuming that a site has k locations and that n_i data points (analyte concentrations) are available for the i^{th} location, the following equations summarize the step-by-step procedure for conducting a parametric ANOVA.

Step 1. Compute the sums and means of each location (i) as follows:

$$X_i = \sum_{j=1}^{n_i} X_{ij}, \Sigma \textit{ of all } n_i \textit{ observations at well } i$$

$$\overline{X} = \frac{X}{N}, \textit{ grand mean of all observations}$$

$$\overline{X_i} = \frac{X_i}{n_i}, \textit{average of all } n_i \textit{ observations at well } i$$

$$X = \sum_{i=1}^{k} \sum_{j=1}^{n_i} X_{ij}, \textit{ grand total of all } n_i \textit{ observations}$$

$$N = \sum_{i=1}^{k} n_i, \textit{ total number of observations}$$

Step 2. Compute the sum of squares of differences between the individual location means and the grand mean:

$$SS_{sample} = \sum_{i=1}^{k} n_i\left(\overline{X}_i - \overline{X}\right)^2 = \sum_{i=1}^{k}\left[\frac{X_i^2}{n_i}\right] - \frac{X^2}{N}$$

This sum of squares has (*k-1*) degrees of freedom associated with it and is a measure of the variability between locations.

Step 3. Compute the corrected total sum of squares

$$SS_{total} = \sum_{i=1}^{k}\sum_{j=1}^{n_i}\left(X_{ij} - \overline{X}\right)^2 = \sum_{i=1}^{k}\sum_{j=1}^{n_i}\left[\left(X_{ij}\right)^2\right] - \frac{X^2}{N}$$

This sum of squares has (*N-1*) degrees of freedom associated with it and is a measure of variability in the whole data set.

Step 4. Compute the sum of squares of differences of observations within locations from the location means. This value is the sum of squares due to error and is obtained by simple subtraction:

$$SS_{Error} = SS_{Total} - SS_{Sample}$$

The sum of squares due to error has associated with it (*N-k*) degrees of freedom and is a measure of the variability within locations.

Step 5. Set up the ANOVA table. The sums of squares and their degree of freedom were obtained from Steps 2 through 4. The mean square quantities are simply obtained by dividing each sum of squares by its corresponding degrees of freedom.

ONE-WAY PARAMETRIC ANOVA TABLE

Source of Variation	Sums of Squares	Degrees of Freedom	Mean Squares	F
Between Locations	$S_{Ssample}$	MS_{Sample}=k-1	SS_{Sample}/(k-1)	F=$MS_{samples}$/MS_{Error}
Error (within Locations)	SS_{Error}	MS_{Error}=N-k	SS_{Error}/(N-k)	
Total	SS_{Total}	N-1		

Step 6. To test the hypothesis of equal means for all *k* locations, compute $F = MS_{Sample}/MS_{Error}$ (last column in above table). Compare this statistic to the tabulated *F* statistic with (*k-1*) and (*N-k*) degrees of freedom (Table 1) at the 5% significance level (*p* level = 0.05). If the calculated *F* value exceeds the tabulated value, reject the hypothesis of equal location means. Otherwise, conclude that there is no significant difference between the concentrations of the *k* locations and thus no evidence of contamination.

Step 7. To determine if the significant *F* is due to differences between upgradient concentrations and downgradient well concentrations a number of post hoc tests can be performed depending on the circumstances including Scheffé test, the Newman-Keuls test, Duncan's multiple range test, Tukey's honest significant difference (HSD) test, and the Bonferroni *t*-statistic. The Bonferroni *t*-statistic, which is commonly used, is described by example in the following paragraphs:

Assume that of the k wells, u are from the upgradient well and *m* are from downgradient wells (i.e., $u + m = k$). Then *m* differences need to be computed and tested for statistical significance. If there are more than five downgradient wells, the individual comparisons are done at the significance level of 1%, which may make the experiments significance level greater than 5% (EPA, 1989).

Step 8. Obtain the total sample size of all upgradient concentrations.

$$n_b = \sum_{i=1}^{u} n_i$$

Step 9. Compute the average concentration from the upgradient well.

$$\overline{X_b} = \frac{1}{n_b} \sum_{i=1}^{u} \overline{X_i}$$

Step 10. Compute the *m* differences between the average concentrations from each downgradient well and the upgradient well.

$$\overline{X_i} - \overline{X_b}, \; i = 1,\ldots,m$$

Step 11. Compute the standard error of each difference as

$$SE_i = \sqrt{MS_{Error}\left(\frac{1}{n_b} + \frac{1}{n_i}\right)}$$

where MS_{Error} is determined from the ANOVA table from Step 5 and n_i is the number of observations at downgradient well *i*.

Step 12. Obtain the *t*-statistic $t = t_{(N-k),(1-\alpha/m)}$ from Bonferroni's *t*-table with α =0.05 and (*N-k*) degrees of freedom.

Step 13. Compute the *m* quantities $D_i = (SE_i)(t)$ for each downgradient well *i*. If $m > 5$, use the entry for $t_{(N-k),(1-0.01)}$. That is, use the entry at $m = 5$.

INTERPRETATION OF BONFERRONI'S STATISTIC:

If the difference exceeds the value $\overline{X}_i - \overline{X}_b$ D_i, conclude that the downgradient well has significantly higher concentrations than the average upgradient concentrations. Otherwise conclude that the downgradient well is not contaminated. This exercise may be performed for each of the *m* downgradient wells individually. The test is designed so that the overall experimental error is 5% if there are no more than five wells.

If the number of wells is greater than five, the experimental error will be greater than 5%. In this case, the Bonferroni *t-test* should be modified by doing the individual comparisons at the 1% level.

NONPARAMETRIC ANOVA

The parametric ANOVA technique is the preferred approach for comparing environmental measurements from downgradient monitoring wells to upgradient well data. However, if the assumption that data sets being compared have the same underlying distribution is violated, non-parametric tests (i.e. Kruskal-Wallis or Wilcoxon Rank-Sum tests) may be used to determine if constituent concentrations present in the downgradient areas significantly exceed those present in the upgradient well.

Non-parametric tests are conducted using the ranks of the analytical results rather than the analytical results themselves. Therefore, the data sets are inspected for extremely high values that may have been underestimated as a result of the ranking process.

The Kruskal-Wallis (EPA, 1989) test should be employed when comparing three or more data sets. However, it is not amenable to two-well comparisons (e.g. one downgradient well to one upgradient well). In these situations, the Wilcoxon Rank-Sum test (EPA, 1992) (also known as the Mann-Whitney U test) should be employed.

The Kruskal-Wallis test is described in the following paragraphs:

Step 1. Rank all N observations of the k ground water wells from least to greatest. As a convention, denote the upgradient well as well 1.

Step 2. Add the ranks of the observations for each well. Call the sum of the ranks for the ith well R_i. Also, calculate the average rank for each well by:

$$\overline{R_i} = \frac{R_i}{n_i} .$$

Step 3. Compute the Kruskal-Wallis statistic given by:

$$H = \left[\frac{12}{N(N+1)} \sum_{i=1}^{k} \frac{R_i^2}{N_i} \right] 3(N+1)$$

where	N	=	total number of samples,
	N_i	=	number of each samples for each well, and
	k	=	number of wells.

An adjustment to the Kruskal-Wallis statistic must be made to account for the presence of tied values. Tied values are those in which have the same concentration for a given analyte. This adjustment is given by the following formula:

$$H' = \frac{H}{1 \left[\dfrac{\sum_{i=1}^{g} t_i^{3} \quad t_i}{N_3 \quad N} \right]}$$

where g is the number of groups of distinct tied observations and t_i is the number of tied observations in the ith group.

Step 4. Compare the calculated value H (or H') to the tabulated X^2 value with (k-1) degrees of freedom, where k is the number of wells

Step 5. If the computed H value exceeds the tabulated X^2 value, compute the critical difference for onsite wells to the upgradient well (denoted as well 1).

$$C_i = Z_{0.01} \sqrt{N(N+1)\frac{1}{12}\left(\frac{1}{n_1} + \frac{1}{n_i}\right)}$$

for i =2,...,k and $Z_{0.01}$ is the upper 90th percentile of the standard normal distribution.

Step 6. Compute the differences between the average ranks of each well and the upgradient well and compare the differences to the critical value from Step 5 in order to determine which onsite wells give evidence of contamination. That is, compare

$$D_i = \overline{R_i} \; \overline{R_1}$$

to C_i for i = 2,...,k. If D_i exceeds C_i , the ith well exhibits statistically significant levels of contamination. If D_i does not exceed C_i , there is no statistically significant evidence of contamination in the ith well.